ABSTRACT

NARANONG, NARAPORN. "Effect of High Energy Radiation on Mechanical Properties of Graphite Fiber Reinforced Composites."
(Under the direction of Dr. RAYMOND E. FORNES.)

This investigation dealt with testing the flexural strength and average modulus of graphite fiber reinforced composites (graphite/epoxy, graphite/polyimide, graphite/polysulfone) before and after exposure to 0.5 Mev electron radiation and 1.33 Mev gamma radiation by using a three-point bending test (ASTM D-790). The irradiation was conducted on vacuum treated samples. The dosages were up to 5000 Mrad for electron radiation and 360 Mrad for gamma radiation. The effect of both types of radiation is to cause a slight increase in the strength and modulus of the graphite reinforced composites.

EFFECT OF HIGH ENERGY RADIATION ON MECHANICAL PROPERTIES OF GRAPHITE FIBER REINFORCED COMPOSITES

By

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BIOGRAPHY

Naraporn Naranong was born [redacted] in [redacted]. She is the first daughter of Mr. and Mrs. Sumedh Naranong. After graduation from Trium-Udom Suksa High School in March 1971, she entered Chulalongkorn University, Bongkok, Thailand. She received a Bachelor of Science Degree in Materials Science in 1975.

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1. INTRODUCTION

Graphite fiber reinforced composites (GFRC) are leading candidates for space structures such as solid fuel rocket motors and hot air ducting [1,2] because of their high strength and modulus, low thermal expansion coefficient and light weight.[2] In space applications, GFRC will be subjected to an environment which includes radiant energy in the form of electrons, protons, ultraviolet and heat, all of which are known to degrade the polymer matrix of the GFRC.[2] Therefore, studies of their responses to high energy radiation have been carried out at North Carolina State University.

The objective of this thesis is to study the effect of the ionization radiation, i.e. electron and gamma radiation, on the breaking strength and the average modulus of the graphite composites, in order to predict the behavior of these composites in the space application.

Several sets of graphite fiber reinforced composites (dimensions 2.54 cm x 1.27 cm x 0.058 cm), fabricated and cut at NASA Langley Research Center in Virgina, were irradiated by 0.5 Mev electron up to 5000 Mrad, or by 1.33 Mev gamma radiation up to 360 Mrad. These were irradiated under a dry, vacuum condition, in order to simulate high energy radiation in the geosynchronous orbits in space and at dose levels that would occur over a thirty-year period.[3] The breaking strength and average modulus of the unirradiated and irradiated samples were determined by using a three-point bending test, according to ASTM D-790. [30] The effect of radiation treatments were analyzed statistically by using polynomial regression.

2. REVIEW OF LITERATURE

2.1 Graphite Fiber Reinforced Composites.

Graphite fiber reinforced plastic (GFRP) is a composite material made of layers of graphite fiber embedded in polymeric matrices such as epoxy resins, thermosetting polymers or thermoplastic polymers and cured using an appropriate temperature and pressure. GFRP is an ideal material used for aerospace structures, components of missile systems, solid-fuel rocket motors, hot-air ducting, fan blades, oven fitting and engine components.[1,2] GFRP is useful in many of these applications because it has very low thermal expansion coefficients and a very high specific strength and modulus.[2] Another advantage is the light weight of the GFRP which reduces cost in space applications when used as substitutes in place of heavy metallic components.

The performance of GFRP depends not only on the strength of the component materials, but also on the coupling properties between fiber and the matrix. In particular, the interaction occurring at the graphite-resin interface determines flexural strength and mode of failure of the composites.[4] The surface of the graphite fibers can be treated in order to improve the interfacial adhesion.[5] Moreover. The elastic properties of composites also depends on their mix ratio and on the orientation of fibers in relation to the direction of a force acting on the composites.[6]

There are many types of graphite fiber reinforced composites with respect to their construction and application. They can be made by laying one or more layers of fibers. The layers can be laid in the same direction or non-parallel. There are many methods to make the

composites; the simplest is the leaky mould technique.[1] A mould to fit the part is made in two halves and the lower section filled with excess of hot-setting phenolic or epoxy resin. Enough wetted fibers are then laid in the mould to fill the moulding cavity when closed. Their orientation is arranged in the direction requiring maximum strength. The upper section of the mould is brought down, squeezing out the excess resin. The resin compound is then cured. The finished composite, which is generally also the finished part, requires only superficial trimming at the end. This method is suitable for making all kinds of simple shapes, but not for complicated forms which have to be made by filament winding or prepreg system. The filament winding involves the creation of a three dimensional shape by winding a continuous length of wetted fiber round and round it. The orientation of the filament at each point is usually designed to bear the load component experienced in use.[1] The prepreg system produces the composite in the form of a raw material which is only later fashioned into the desired shape. The raw material is made in the form of a uniform sheet or tape known as "warp," oriented fiber or "prepreg." Prepreg is made by dipping numerous tows or groups of fibers in a dilute solution of resin in acetone and then laying them down parallel and without overlap, on a firm flat surface, which should not react with the fiber or resin in anyway. A second sheet of this material is placed on top, the assembly is warm rolled to even thickness, eliminating voids between fibers and dried in the usual way. All carbon fibers in bulk-use are marketed in partially cured prepreg form except in special case.[1] There are some problems associated with fiber matrix adhesion in carbon composites. In the case of

a composite made with a thermosetting resin, the fiber-matrix bond strength is improved by processing the carbon fiber surface.[1]

2.2 Graphite

Graphite is one of three allotropic forms of carbon found in nature: amorphous, graphite and diamond. Graphite exists in two forms: alpha and beta.[7,8] The two forms have very similar physical properties except for their crystal structures. The alpha form has hexagonal crystal structure with lattice parameter: a = 2.46 Å, c = 6.71 Å [9] and the beta form has rhombohedral crystal structure with lattice parameter: a = 2.65 Å, α = 55.3°.[9] Naturally occurring graphite is reported to contain as much as 30% of the rhombohedral (beta) form, whereas synthetic materials contain only the hexagonal (alpha) form. The alpha form can be converted to the beta form by mechanical treatment and the beta form reverts to the alpha form on heating above 1000°c.[8] Natural graphite has a specific gravity of 1.9 to 2.3.[8] The now accepted graphite structure is shown in Figure 1.[7] In this stable hexagonal lattice the nearest interatomic distance within a layer plane is 1.415Å and 3.354Å between basal planes. Covalent bonding force exists within the layer planes and the weak Van der Waals force exists between planes. The bonding energy between planes is only about 2% of that within planes. The weak forces between layer planes account for:

(a) The tendency of graphite to fracture along a plane.

(b) The formation of interstitial compounds.

(c) The lubricating, compressive and many other properties of graphites.[7]

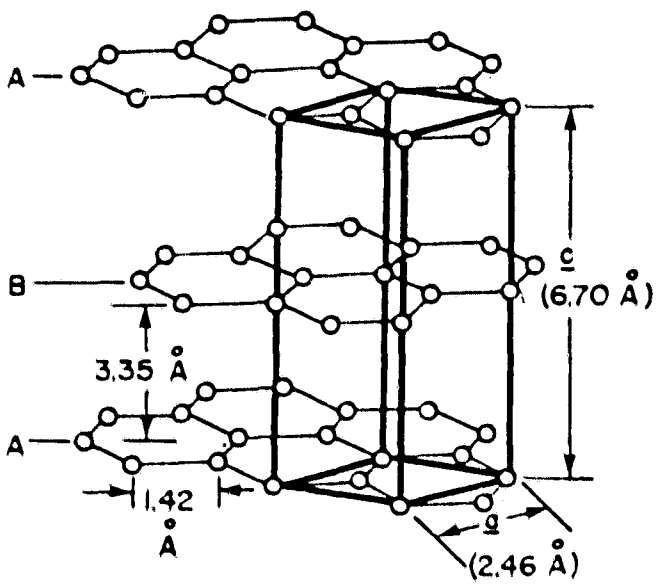


Figure 1. Graphite crystal structure.

Graphite or carbon fiber has a very high modulus e.g., Thornel 300 has the modulus of 33.2 x 10^6 p.s.i. (2.3 x 10^6 kg/cm^2) and the breaking strength of 408 x 10^3 p.s.i. (2.8 x 10^4 kg/cm^2).[10] Carbon fiber can be produced by carbonizing (heating at 1000° in an inert gas) organic precusor fibers, and then graphitizing them at a very high temperature, above 2500°c.[11] The most widely used precusor fibers are polyacrylonitril (PAN) and rayon fibers, but a number of other precusors such as polyvinylalcohol, polyimide, phenolics and pitches have also been used.[11] The densities of carbon fibers prepared from rayon and PAN are ≈ 1.6 g/cm^2 and ≈ 1.9 g/cm^3 respectively.[11]

2.3 Polymeric Matrices

2.3.1 Epoxy

Epoxy is a thermosetting polymer based on a polyether. They may be formed in condensation reactions such as reaction between bisphenol A and epichlorohydrin. Relatively large portions of curing agents, most of which are amines, are required to cure apoxy.[12] The high concentration of hydroxy and other polar groups in the molecular structure of the cured apoxy results in very strong adhesive forces.

The epoxy used in this study is NARMCO 5208, which is composed of tetraglycidyl -4,4'- diaminodiphenyl methane (TGDDM) cured with 4,4'-diamino diphenyl sulfone (DDS).

Morgan and O'Neal [13] stated that the crosslinked network structure, the microvoid characteristics, the fabrication stress and the environmental factors are the major structural parameters controlling the mode of deformation and failure and the mechanical response of the TGDDM-DDS epoxies. They reported the gradual decrease in tensile

strength and modulus and the increase in ultimate elongation as the test temperature was elevated to 200-250°C, which suggested that a broad glass transition (T_g) exist in the temperature range. The sorption of moisture by epoxies lowered their T_g and the microscopic yield stress.[13] The sorbed moisture caused epoxies to swell. This swelling stress together with other stress inherent in the materials, could be sufficiently large to cause localized fracture of the polymer.[13]

2.3.2 Polyimide

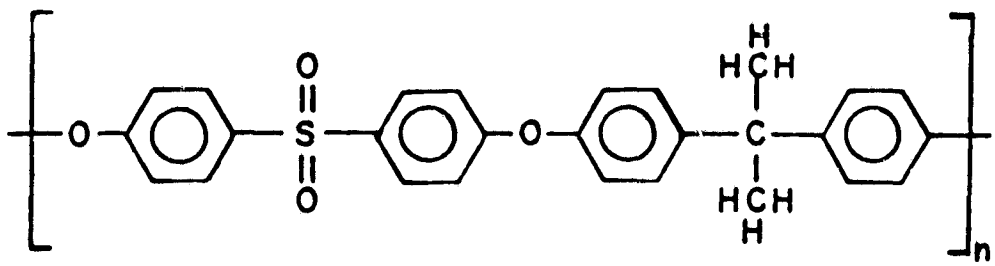
Polyimides are thermosetting polymers, which are commonly prepared from dianhydrides and diamines with pyrometallic dianhydrides.[14] The reaction involves initial formation of polyamide followed by ring closing to form polyimide. A matrix used in this study is PMR-15 which is a thermosetting polyimide developed by NASA, manufactured by Ciba-Geigy Corporation.[15] This polyimide resin powder was prepared from the precursor constituents, 5-norbornene -2,3 - dicarboxylic anhydride (NBA), 3,3',4,4' - benzo-phenone tetra carboxylic dianhydride (BTDA) and 4,4', methylene dianilene (MDA) which were obtained from Eastman Organic Chemicals.

The cured PMR-15 matrix showed a significant decrease in fracture energy when tested at 350°C which is its T_g.[15] Hexcel F-178, another thermosetting polyimide showed a marked decrease in fracture energy, i.e. embrittlement, caused by thermal aging.[15] Hexcel F-178 can be cured by electron radiation (3MeV, 20 Megarad) and have many of the properties desired for high-performance [15] applications.

2.3.3 Polysulfones

The polysulfone matrix used in this study is P1700 (UDEL-P1700, by Union Carbide Corporation), which is the aromatic polysulfone poly(oxy-1,4-phenylene sulfonyl-1,4-phenylene oxy-1,4-phenylene isopropylidene-1,4-phenylene).[16] The specimen can be prepared by melting pellets of the thermoplastic polysulfone in an open aluminum dish.[15] P1700 is a high strength and modulus material which retains these properties after exposure to high temperature and high energy radiation.[15] Its glass transition temperature is reported to be 174°C [15] and 190°C.[16]

The structure of polysulfone[16] is shown below.



2.4 High Energy Radation and Its Interaction with Matter

The term high energy radiation is applied both to particles moving with high velocity, i.e., fast electron or β-particles, fast protons, neutrons and α-particles, and to electromagnetic radiation of short wavelength, i.e., x-rays and gamma rays.[17] In the latter, the radiation can best be considered as a series of individual particles (photons) each of high energy. The energy carried by each particle or photon is very much greater than that binding any orbital electron to an atomic nucleus. In passing through matter, all these forms of high energy radiation lose energy by reacting with the orbital electrons and nuclei of the medium. This may give rise to displaced nuclei, free

electrons, ionized atoms or molecules and excited atoms or molecules (in which an electron is raised to a higher energy level).[17]

2.4.1 Electron Radiation

In passing through a specimen, each electron loses most of its energy by interaction with the orbital electrons. The primary electron is deviated, and a bound electron may either be given sufficient energy to leave the parent atom (ionization) or move to an orbit of higher energy (excitation). Ionization results in a positively charged atom or molecule (ion) and a free electron. The positive ion is unstable and may undergo decomposition or reaction with neighboring molecules or other ions. The free electrons may either return to the parent atom to give a highly excited molecule or it may be captured elsewhere giving a negative ion.[17]

The net effect of the high energy electrons is to cause non-uniform ionization up to the maximum penetration of the primary electrons. For incident electrons of 1 Mev or above, the maximum penetration in water is approximately 0.5-0.6 cm per Mev.[17] For materials of different density the penetration can best be expressed in g/cm^2, since for most atoms the density of electrons (which determines the ionization) is approximately proportional to the atomic weight. The following formula gives the penetration R (in g/cm^2) in aluminum of β-rays of maximum energy, E (in Mev), between 0.7 and 15 Mev [17]:

$$R = 0.543\ E - 0.160$$

For relativistic velocities the energy loss per centimeter of high energy electron is given as:[18]

$$\frac{-dE}{dx} = \frac{2\pi Ne^4}{mv^2} Z \left\{ \log \frac{mv^2 E}{2 I^2 (1-\beta^2)} - (2\sqrt{1-\beta^2} - 1 + \beta^2) \log 2 \right.$$

$$\left. + 1 - \beta^2 + \frac{1}{8} (1 - \sqrt{1-\beta^2})^2 \right\}$$

where

E is the kinetic energy of the incident electron.

x is the distance into the scatter.

N_i is the member of the i^{th} type atoms in the scatter.

Z_i is the atomic number of the i^{th} type atom.

e is the electron charge.

I_i is the average ionization potential of the i^{th} atom.

m is the electron rest mass.

v is the incident electron velocity.

$\beta = \frac{v}{C}$

C is the speed of light in vacuum

2.4.2 Gamma Radiation

In passing through matter, gamma rays may lose energy by colliding with the orbital electrons (Compton effect) causing ionization, by photoelectric absorption, by reacting with the nucleus to form a radioactive isotope or pair production. In water the photoelectric effect is the major process below about 60 kev, and Compton scattering predominates at energies between 60 kev and 25 Mev. Pair production can occur only at energies above 1.02 Mev and in water

is the major effect only above 25 Mev. The gamma radiation used in this study has energy of about 1.33 Mev; therefore, the major energy loss is by Compton scattering which causes ionization.

The loss of energy by Compton scattering arises from elastic collision between the photon and an electron of the medium.[17] Gamma photons with energy of 0.3 to 1 Mev will transfer about 3% of their energy per g/cm^2 to electrons, which will then cause further ionization and excitation.

Pair production of an electron and a positron by a photon can only occur at photon energies exceeding 1.02 Mev equivalent to twice the rest mass of an electron. The presence of an atomic nucleus is also necessary to carry off excess momentum. In the radiation work here the effect of pair production is negligible.[17]

Photoelectric absorption occurs when a gamma, photon or x-ray photon of low energy, of the order of a few kev for atoms of low molecular weight, is absorbed by the inner electrons of an atom and the electron ejected. The electrons ejected by the photoelectric effect lose their energy by ionization and excitation of neighboring molecules.

2.4.3 Fast-Neutron Radiation

Fast neutrons, being uncharged, do not react with orbital electrons but lose their energy primarily by elastic collisions with atomic nuclei.[17] In a fast neutron collision all the neutron energy may be transferred to a hydrogen atom, producing a high energy proton. For a carbon atom the maximum amount of energy which can be transferred is only 28%. In most polymers, hydrogen constitutes the largest number of atoms present, and the main effect of fast neutron irradiation is the

production of fast protons within specimen. These protons have a very short range, but cause the intense local ionization and excitation.[17] For the same energy absorption, the changes resulting from neutron irradiation may be different from those due to electron or γ-radiation.[17]

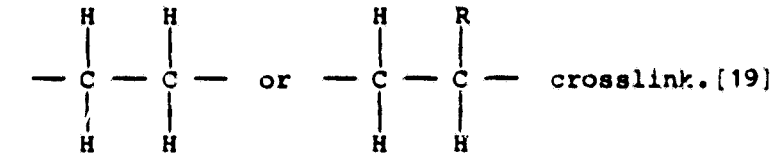
A fast neutron flux of 10^{16} neutrons/cm^2 of average energy 1 Mev will lose about 1.96×10^{21} ev in passing through 1 g/cm^2 of polyethylene. The corresponding dose is 32 Mrad. In other polymers such as polytetrafluoroethylene, it is considerably less.[17]

Neutrons with low kinetic energy of the order of thermal energies (0.025 ev) have insufficient energy to cause direct ionization and excitation.[17]

It has been repeatedly confirmed that the major reaction in polymers, whether produced by fast electron, x-rays or gamma rays, or mixed radiation including neutrons from atomic reactor, depends primarily on the total energy absorbed and sometimes on the radiation intensity, but rarely, if ever, on the type of radiation or its sources.[19]

2.4.4 Crosslinking and Chain Scission

In most long chain polymers, the major effect of exposure to high energy radiation is either the fracture of side chains leading to the formation of crosslinking or alternatively the fracture or scission of main chain bonds, resulting in a reduction in average molecular weight.[17] Polymers containing divinyl units of the form:



[19]

It has been confirmed that polymers which show degradation suffer main chain fracture only (no crosslinking) and that the observed reduction in molecular weight is not due to competition between the two processes of crosslinking and degradation, the latter predominating.[19] In the case of crosslinking polymers, it has not yet been discovered whether some degree of degradation also takes place.[19]

Aromatic compounds have the special property that they are resistant to radiation. Parkinson and Sisman [12] explained this property of polymers. For example, the phenyl ring in polystyrene stabilizes it from radiation damage. The resonance-stabilized aromatic ring can absorb energy by going to an excited state and can dissipate this excitation energy through a process which does not disrupt the molecule. They concluded that aromatic compounds can dissipate excitation energy without decomposition.[12]

2.5 Effect of High Energy Radiation of Graphite Fiber Composites

Since graphite reinforced composites are leading candidates for materials in space structures, we need to know and understand their behavior in a space environment. Space environment includes radiant energy in the form of electrons, protons, ultraviolet radiation and thermal radiation, all of which are known to degrade mechanical and physical properties of the polymer matrix in the composites.[2]

Therefore, research on the durability of the graphite composites in a space environment is needed.

There are some important conclusions of the Assessment Committee of the need for NASA research on the durability of materials in a space environment which can be summarized below: [2]

(1) The radiation damage mechanisms are not understood well enough to be modeled for analytical prediction of degradation of composites. The mechanism of the fundamental radiation induced processes, crosslinking and scission, are probably known for the simplest plastics and elastomers, but for the chemically complex, heavily reinforced materials, even the basic degradation processes are unknown.

(2) Electrons and protons do not produce the same degradation, both in terms of dosage effects and damage depth profile effects. Electrons have far greater penetration than protons and will affect the bulk properties of materials. Protons will affect mainly the surface.

(3) There are many open literature publications about ionizing radiation effects on the epoxy system over the past 20 years. However, only a few of these studies involved epoxy/carbon fiber composites, and much further information concerning high dose/mixed radiation effects on engineering properties is needed.

In this literature review, the effects of gamma radiation, electron radiation and some other types of radiation, such as neutron, on composites, graphite fiber and the matrices will be discussed, respectively.

2.5.1 Effect of Gamma Radiation

2.5.1.1 Graphite Reinforced Composites

Lackman, et al,[20] in 1971, exposed unidirectional graphite epoxy composites (AS/3002) to nuclear radiation (neutron and gamma) up to 2.6 Mrad. They concluded that there was no degradation in longitudinal flexural strength, transverse strength and horizontal shear strength.

In 1972, Bullock, et al[21] studied the tensile strength of graphite-epoxy composites after they were irradiated for 600 hours in air at ambient temperature, below 75°C, with a Ground Test Reactor (GTR) operating at a power level of 10 MW. In two experiments, the gamma radiation doses were calculated by taking the ratio of gamma dose to fast neutron fluence from the mapping irradiation and was determined to be 2.7×10^{11} ergs/g (2700 Mrads) and 5.8×10^{11} ergs/g (5800 Mrads), respectively. Their conclusion was that no effect of radiation was observed on tensile strength of graphite-epoxy composites.

R. E. Bullock[22] also irradiated unidirectional graphite-epoxy composites (HT-S*/ERLA 4617) with the GTR in air at ambient temperature below 75°C and in liquid nitrogen (LN_2) at -196°C. The longitudinal flexural strength of specimens irradiated in air to 2700 Mrad and tested at ambient temperature was 20% lower than that of the unirradiated specimen, while the transverse strength of these specimens was 85% lower than the control value. Then, with additional radiation exposure (to 5800 Mrad), the longitudinal flexural strength decreased by more than 70%, but the transverse strength changed very little. Thus, it appeared that only moderate transverse flexural

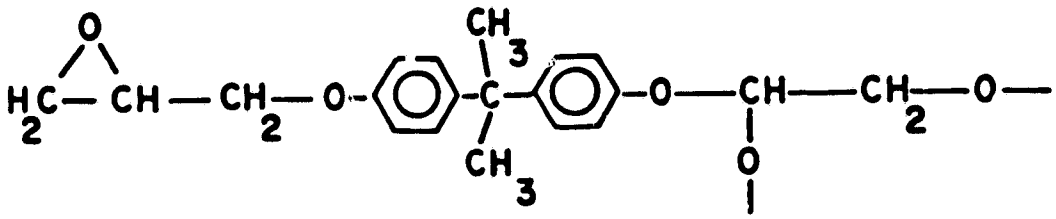
strength was required to develop good translation of fiber strength into longitudinal strength, and the strength of the matrix itself did not seem to be nearly so critical as does the strength of its bonding with fiber reinforcements. However, the longitudinal flexural strength increased 80% after exposure to 8.9 x 10⁷ ergs/g (8900 megarad) of gamma dose in liquid nitrogen (LN_2) at -196°C and tested at -196°C.[22] Bullock concluded that the increase was probably caused by a radiation induced lowering of the too-high interlaminar shear strength of the specimens in LN_2 before irradiation.

In 1978, Arrington and Harris [23] studied the effect of exposure to steam and to gamma radiation in order to provide information on possible environmental effects. They used unidirectional carbon fibers aligned in an epoxy matrix and used a three-point bending tester with a span length of 13 mm to measure the interlaminar shear strength. Their experimental results showed that there was a slight enhancement of the strength by radiation treatment, but the average work of fracture was reduced. They suggested that radiation increase the matrix crosslink density and stiffen it.

2.5.1.2 Matrix Materials

(a) Epoxy Matrix

Epoxy polymers are thermosetting resins based on polyethers. For example, they may form in a condensation reaction between bisphenol A and epichlorohydrin. They require relatively large portions of curing agents. The most common curing agents are amines, thereby nitrogen is usually incorporated into the molecular structure.[12]



Parkinson and Sisman[12] explained that the phenyl rings stabilized the polymer against radiation by absorbing energy in going to an excited state and dissipating this excitation energy through a process which does not disrupt the molecule. Therefore, the radiation resistance of epoxies is fairly good. Their work on effect of radiation on flexural strength of the epoxies showed that the aromatic cured epoxies had much more resistance than those obtained with aliphatic curing agents. For example, the aromatic amine-cured epoxy (diamino diphenyl methane) retained >80% of initial its strength when irradiated in air with gamma radiation and neutrons up to 1000 Mrad from a nuclear reactor. The aliphatic amine-cured epoxy (piperidine) retained 80-100% of its strength at a gamma doses of ≈200 Mrad, and lost its strength to 10-50% of initial value when the doses gradually increased to 1000 Mrad. The acid anhydride-cured epoxy (hexahydrophthalic anhydride) retained 80-100% of its initial strength at ≈600 Mrad, retained 50-60% of its strength at 600-1000 Mrad, and lost strength to 10-50% of its initial value beyond 1000 Mrad.[12]

The epoxy matrix used in this study, NARMCO 5208, (tetraglycidyl diamino diphenyl methane cured with diaminodiphenyl sulfone), which is an aromatic epoxy, shows good radiation resistance as shown in the experiments that we report here.

Parkinson and Sisman's[12] radiation work was done in air. They stated that oxygen played an important part in degrading the epoxies. However, the irradiation in this study is done under vacuum conditions so less degradation should be expected.

(b) Polyimide Matrix

The polyimides are condensation polymers of the anhydrides of tetracarboxylic acids and primary diamines.[12] Polyimides are relatively resistant to radiation. Little change occurred in strength after irradiation of thick specimens to 10^{10} rad. (10,000 Mrad).[12] But a 0.002-in. film irradiated in air at ~2 x 10^6 rad/hr. lost over half their initial tensile strength at 10^{10} rad. (10,000 Mrad), and the elongation at break was less than 10% of the initial value. However, the film irradiated in vacuum retained its tensile strength up to 10^{10} rad. and retained the elongation at break up to 5000 Mrad. It decreased to 50-80% of initial value of elongation-at-break at radiation doses from 5000 to 10,000 Mrad.[12]

(c) Polysulfone Matrix

The molecular chain of polysulfone consists of phenylene rings linked alternately by quaternary carbon atoms, oxygen and sulfone groups. The phenylene ring and the sulfone group confer resonance stabilization, but the quaternary carbon in the chain leads to scission. Radiation stability would be expected to be only moderate.[12]

In 1979, Brown and O'Donnel's[16] work on P1700, the aromatic polysulfone investigated in this study, showed no change in flexural yield strength (determined according to ASTM D-790) after a gamma dose up to 600 Mrad, irradiated in vacuum at 35°C and 80°C, but at

125°C the strength was significantly higher (15%) than the value for the unirradiated samples (note Tg = 190°C). However, the flexural strength decreased to 40-60% of the initial value with doses of 200-400 Mrad on irradiation in air, depending on the samples and irradiation conditions. They suggested that it could result from a decrease in molecular weight at the surface of the test specimens due to radiation-oxidation reaction. Since glassy polymers usually show a sigmoidal strength-molecular weight relationship, the decrease in strength results from the reduction in molecular weight. According to Brown and O'Donnell, the most suitable method for expressing quantitatively the effect of radiation on the molecular weight of a polymer is to calculate the yield of main-chain scission [G(s)] and crosslinking [G(x)], which can be calculated from measurements of the soluble fraction, s, of the polymer after various radiation doses, in excess of the gel dose. They concluded that chain crosslinking was predominant over scission for irradiation in vacuum at all temperatures (below Tg = 190°C).

2.5.2 Effect of Electron Radiation

Brown, et al,[36] studied the tensile strength of graphite fiber reinforced epoxy laminates and unreinforced epoxy resin after being exposed to 300 kev electron radiation in vacuum. They reported that there were no significant change in the modulus and ultimate strengths of the two materials tested after up to 10 years simulated exposure.

2.5.3 Effect of Other Types of Radiation

2.5.3.1 Graphite Reinforced Composite

Kibler, et al[24] studied the physical response of graphite-epoxy composite (T300/5208) to continuous wave CO_2 laser radiation, and made comparisons with aluminum (2024(T81)) composites. They used the term "beam intensity" to classify the strength of the laser beam, which they defined as $I = P/\pi r^2$. (I is beam intensity or power density in w/cm^2, P is the laser power in watts, r is the beam radius, as measured by ablation of plexiglass, in cm.)

At intermediate intensities ($I \approx 1500$ w/cm^2) the strength retention for laser damaged graphite-epoxy composites and aluminum were similar. At high CO_2 laser intensity ($1500 < I < 6400$ w/cm^2), aluminum was more easily penetrated than graphite epoxy, and the strength retention of aluminum was significantly less than graphite epoxy composites.

Bullock[25] found that the strength of the graphite/epoxy composites made up with fast-neutron-irradiated graphite fibers was higher than the composite made up with the unirradiated graphite fibers. In addition, the interlaminar shear strength, an indicator of fiber-to-resin bonding, was somewhat improved for the composite made up with irradiated fibers.[25]

Bullock[22] also found that the strength of the graphite epoxy composites increased as much as 80% when irradiated by fast neutron ($E > 1$ MeV, fluences 5.7×10^{17} n/cm^2).

2.5.3.2 Matrix Materials

Parkinson and Sisman's[12] work on the radiation effect on the polymer, e.g., epoxy, polyimide, polysulfone, was carried

out by using gamma radiation and neutrons from a nuclear reactor. They also reported that the interaction of both gamma radiation and fast-neutrons with matter yielded energetic particles, which interacted with the orbital electrons of the atoms of the absorbing medium to ionize or excite them to higher energy levels.

Section 2.5.1.2 will also provide some information about the effect of fast-neutron radiation on the polymer matrices.

2.5.3.3 The Graphite Fiber

Graphite under neutron irradiation exhibits significant creep and dimensional change, both of which are dependent on the accumulated dose and temperature.[35]

In 1973, McKague, et al[26] reported that fast neutron irradiation induced increases in the tensile strengths and elastic moduli of carbon fibers and these property improvements translate largely into composites reinforced with such fibers. Moreover, as an added benefit, the fiber-to-matrix bonding was improved in irradiated fiber composites. They concluded that neutron irradiation caused change in the crystalline structure of carbon fibers and improved the strength of the fiber-matrix interface.

In 1972, Jones and Peggs[27] reported a 40% increase in average fracture strength and a 12% increase in Young's modulus of graphite fibers after irradiation by a fast-neutron dose of 6.6×10^{20} n/cm^2 at 550 ± 10°C. However, they also noted that the maximum and minimum values of fiber density and apparent crystallite size coincide with the maximum and minimum values of strength and

Young's modulus. (The increase in crystallite dimension may be due to annealing.)

Bullock[25] reported that the tensile strength of the graphite fibers was very stable under fast-neutron radiation exposure, where large, rapid changes in the temperature do not occur. The graphite fiber was strengthened as much as 30% when irradiated beyond 8.5×10^{17} n/cm^2 in an inert environment. On the other hand, the tensile strength of the graphite fiber was subject to severe oxidation degradation when irradiated in air at ambient temperature (< 80°C).

Gray[28] reported the dimensional change of eleven different types of carbon and graphite fibers which had been exposed to fast neutron radiation to the fluences of 3.5, 7.3 and 10×10^{21} n/cm^{-2} at 470°C. The axial shrinkage was 18 to 27%, the radial shrinkage was 19 to 33%. This effect also happened in graphite cloths, the amount of the dimensional change depended on the types of weaves. The 2-dimensional cloths remained essentially unchanged. On the other hand, the 3-dimensional cloths deteriorated apparently because this type of weave was less able to accommodate the large axial fiber shrinkage.

Reynolds[29] referred to Baker and Kelly's work in which they concluded that an irradiation dose of 4×10^{17} neutron/cm^2 at room temperature raised the effective shear modulus of the graphite crystal (S_{44}), from 5×10^8 dynes/cm^2 to over 1.6×10^{10} dynes/cm^2. Using a transmission electron microscope, Baker and Kelly were able to show that the change in S_{44} both on irradiation and on subsequent annealing correlated with the observed pinning of basal-plane dislocation by submicroscopic defects.

Kelly also irradiated several different types of graphite at 170°C under similar conditions. He found that they all showed a Young's modulus decrease of about the same amount at each dose and concluded that the shear modulus of the crystallites must decrease with increasing c-axis spacing. This effect is not yet understood.[29]

Reynolds[29] tried to establish a complete theory of irradiation damage in graphite, which would provide the prediction of the overall atomic redistribution due to exposure of graphitized material at any temperature to a given neutron flux. However, he was unable to establish a successful model.

3. EXPERIMENTAL PROCEDURE

Four types of the unidirectional graphite fiber reinforced composites (GFRC) were used in this study. The GFRC specimens, ca. 2.54 cm x 1.27 cm x 0.058 cm size, were 4-ply, uniaxially aligned along the 2.54 cm length and were fabricated, cured and cut at NASA Langley Research Center, Hampton, Virginia. Several sets of these GFRC specimens have been irradiated using 0.5 Mev electron radiation or 1.33 Mev gamma radiation under pre-vacuum or continuous vacuum conditions. The effects of irradiation treatments on the breaking stress and Young's modulus were determined by a three-point bending tester, attached to an Instron testing machine, according to ASTM D-790.[30]

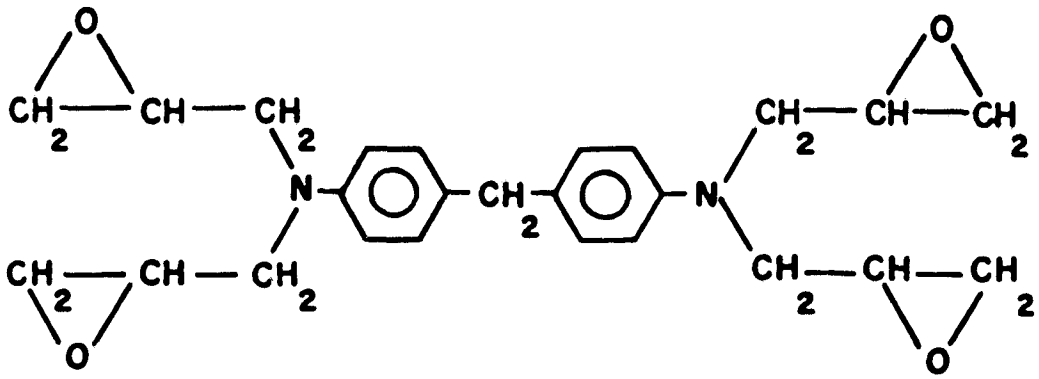
The load and deflection were recorded on graph paper. The breaking stress and Young's modulus were calculated and plotted against irradiation dose. The results were analyzed using polynomial regression models and the Duncan's Multiple Range Comparison (which is less informative than the polynomial regression).

3.1 Materials

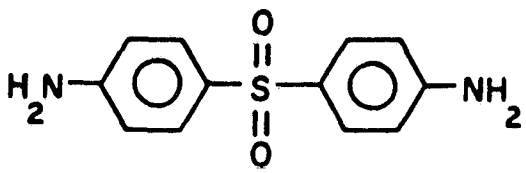
3.1.1 T300/5208

This graphite/epoxy composite is made of Thornel 300 (graphite fiber by Union Carbide Corporation) impregnated in the NARMCO 5208 (epoxy resin by Narmco Materials, Inc.). Thornel 300 is a multi-filament strand of three thousand filaments, each filament having a 7 μm diameter.[10] It has a high elastic modulus of 33.2 x 10^6 p.s.i. (2.3 x 10^6 kg/cm^2) and breaking strength of 408 x 10^3 p.s.i. (2.8 x 10^4 kg/cm^2).[10] NARMCO 5208 is composed of tetraglycidyl -4,4'-diamino diphenyl methane (TGDDM) cured with 4,4'-diamino diphenyl sulfone

(DDS).[15] The structures of the epoxy resin and the curing agent are shown below:[13]



Tetraglycidyl -4,4'-diamino diphenyl methane (TGDDM)



4,4'-diamino diphenyl sulfone (DDS)

The glass transition temperature of NARMCO 5208 is reported to be 196°C[37] and 260°C[15].

Two batches (A and B) of samples, fabricated at two different times at NASA Langley Research Center, were investigated. Approximately 130 specimens from batch A were sent and 80 specimens were used for electron radiation studies and 50 specimens from this batch were used for gamma radiation studies. Approximately 100 specimens from batch B were sent and used only for electron radiation studies.

The first electron radiation treatments were at 0, 10, 50, 100, 200, 400, 800, Mrad. Eleven specimens from batch A were used at each treatment. The gamma radiation treatments were at 0, 40, 83, 167 Mrad. Approximately 10 specimens from batch A were used at each treatment. The second electron radiation treatments were at 0, 100, 200, 300, 500,

1000, 2000, 3000, 4000, 5000 Mrad. Approximately 10 specimens were used at each treatment.

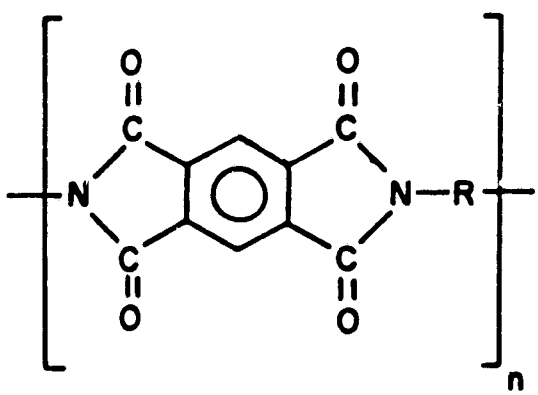
One batch (100 specimens) of sampes were used in gamma radiation studies at 0, 40, 83, 167, 357 Mrad, 20 specimens were used at each radiation treatment.

3.1.2 AS/3501-6

Both the epoxy resin 3501 and the graphite fiber AS are manufactured by Hercules Corporation.

3.1.3 C6000/PMR15

This graphite/polyimide composite is made of the Celion graphite fiber by Celanese Corporation, impregnated in PMR-15 polyimide matrix, developed by NASA and manufactured by Ciba Geigy Corporation.[15] The general structure of polyimide is shown below:



POLYIMIDE

PMR15, a thermosetting polyimide resin powder, is prepared from the precusor constituents 5-norbonene -2,3-dicarboxylic anhydride (NBA), 3,3', 4,4'-benzophenone tetracarboxylic dianhydride (BTDA) and 4,4'-methylene dianilene (MDA) which were obtained from Eastman Organic Chemicals. The NBA (98 gm) and BTDA (201 gm) were dissolved in methanol (190 cm^3) by refluxing for one hour. The MDA (183 gm) dissolved in

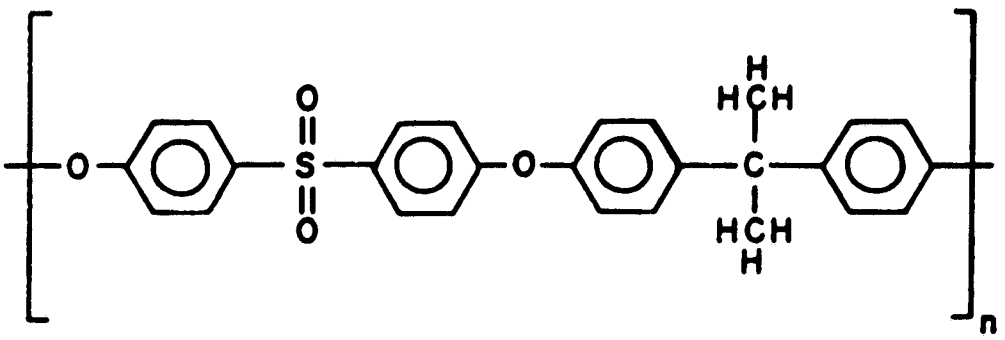
methanol was added to the mixture of the anhydrides. The majority of the methanol was removed by vacuum evaporation at low temperature (30 - 40°C) to give a viscous syrup. The remaining alchol was removed by placing the syrup in aluminum pans and heating at 130 - 145°C for one hour in an evacuated (5 mm Hg) oven to form a glassy foam. This polyimide resin should be post-cured at 316°C for 16 hours to form the cured matrix.[15]

The glass transition temperature of PMR15 is 350°C.[15]

Two batches (A and B) of samples were investigated. Approximately 91 specimens of batch A were used in electron radiation studies at various doses, i.e. at 0, 10, 50, 100, 200, 400, 800 Mrad, 13 specimens were used for each radiation treatment. Approximately 110 specimens of batch B were used in electron radiation studies at 0, 100, 200, 300, 500, 1000, 2000, 3000, 4000, 5000 Mrad, 11 specimens were used for each radiation treatment.

3.1.4 C6000/P1700

This graphite/polysulfone composite is made of Celion graphite fiber by Celanese, impregnated in P1700, polysulfone resin manufactured by Union Carbide Corporation.[15] P1700 is prepared by melting pellets of the thermoplastic polysulfone in an open aluminum dish.[15] P1700 is an aromatic polysulfone, consisting of poly(oxy-1,4-phenylene-sulfonyl-1,4-phenyleneoxy-1,4-phenyleneisopropylidene-1, 4-phenylene).[16] The structure of P1700 is shown below:[16]



POLYSULFONE

The glass transition temperature is reported to be 174°C [15] and 190°C.[16]

One batch of sample were investigated. Approximately 100 specimens were used for electron raidiation studies at various doese, i.e. 0, 10, 200, 300, 500, 1000, 2000, 3000, 4000, 5000 Mrad and 10 specimens for each treatment.

3.2 Equipment

3.2.1 Radiation Equipment

Radiation from an electron accelerator and Cobalt-60 gamma source were used in this study. The electron accelerator, manufactured by High Voltage Engineering Corporation, was operated at 8.3 milliamperes beam current and 500,000 volts (from an insulated core transformer). This equipment utilized a horizontal beam scanned to 48" by 6". The samples were hung vertically on a conveyor, which carried them in front of the beam twice in each revolution through the equipment so that the samples received half of their total dose from each side. All irradiations were carried out in nitrogen-filled ziploc polyethylene bags from Dow Chemical Corporation. Radiochromic nylon films with incorporated aminotriphenyl methane dyes derivative made by Far West

Technology, Inc. were used for dosimetry measurements. These films became colored on radiation and the radiation dose could be determined from a calibrated curve giving optical density. The calibrated curves were made using the Radiochromic Reader, model 91-R, also from Far West Technology, Inc.

The 1.33 Mev gamma radiation was obtained from the Gamma Cell 220 colbalt-60 source with a known dose rate of approximately 0.33 Mrad per hour.

3.2.2 Strength Testing Equipment

3.2.2.1 Composites

The ultimate stress and the average modulus of the composites were determined from flexural strength deformation curves by using a three-point bending tester, attached to the Instron testing machine, according to ASTM-D790[30] and ASTM STP 674.[31] A compression load cell with maximum load range 200 pounds and the crosshead speed of 0.254 cm/min were used (see Figures 2, 3a, 3b). With a three-point bending test, the load was applied perpendicular to the plane of the composite with a spanlength of 1.40 cm. The specimen were 4-ply, uniaxially oriented with the preferred axis aligned along the span direction during testing. The breaking load and the deflection were recorded and used to calculate ultimate stress and average modulus, using the formulas stated in Section 3.4.

3.2.2.2 Graphite Fibers

Tensile strength of the graphite fibers bundles were determined using the Instron testing machine with the crosshead speed 0.05 cm/min (0.02 in/min) and with 2.54 cm-gauge length. Details

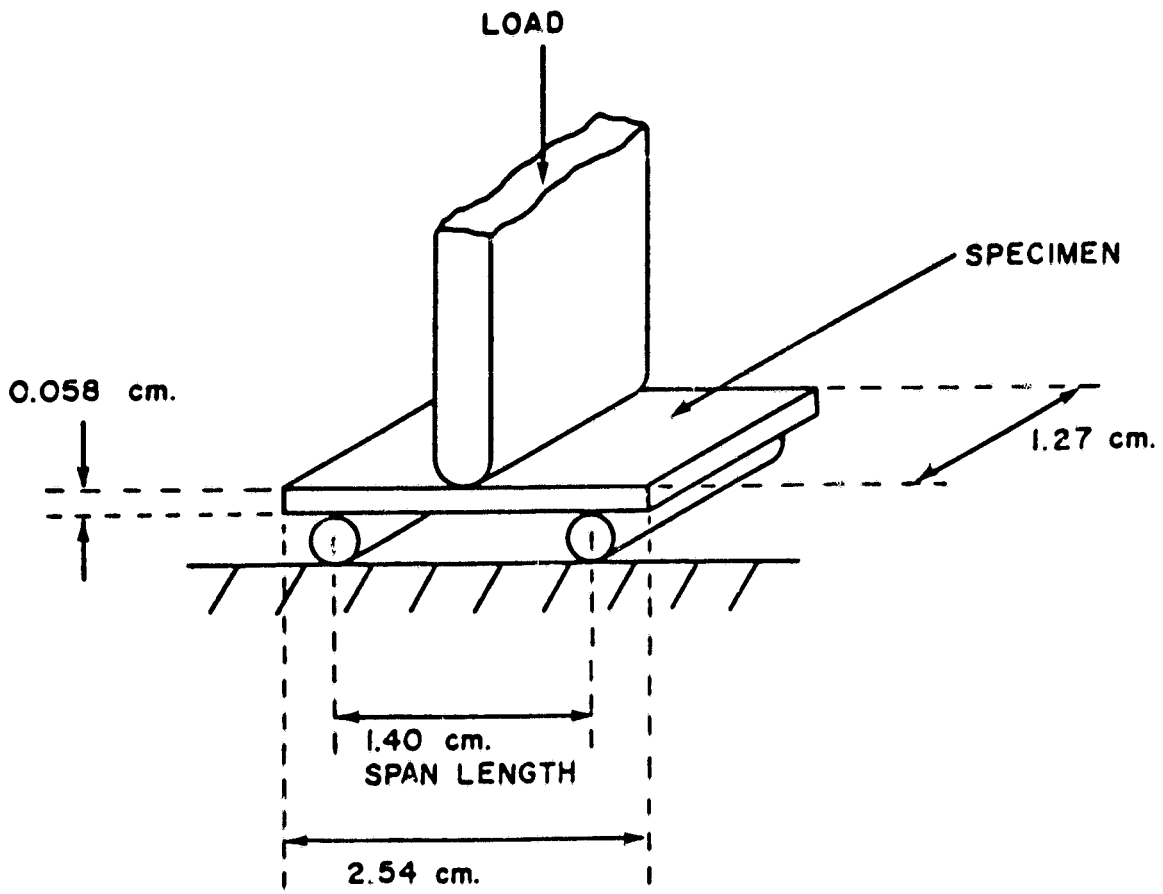


Figure 2. Diagram of a three-point bending tester, with a specimen in place.

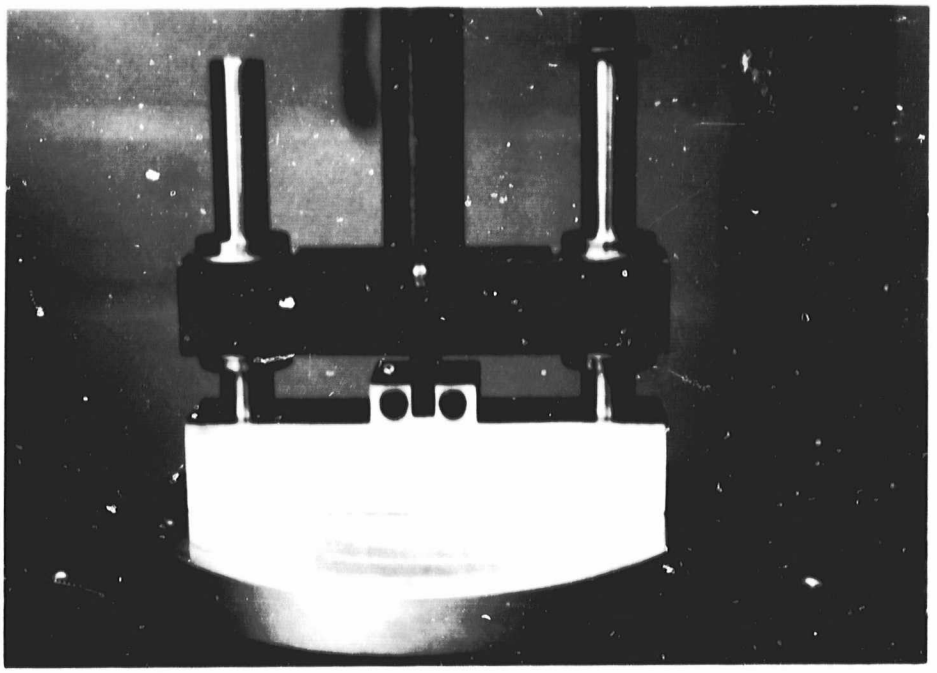


Figure 3. a. Picture of a three-point bending tester.

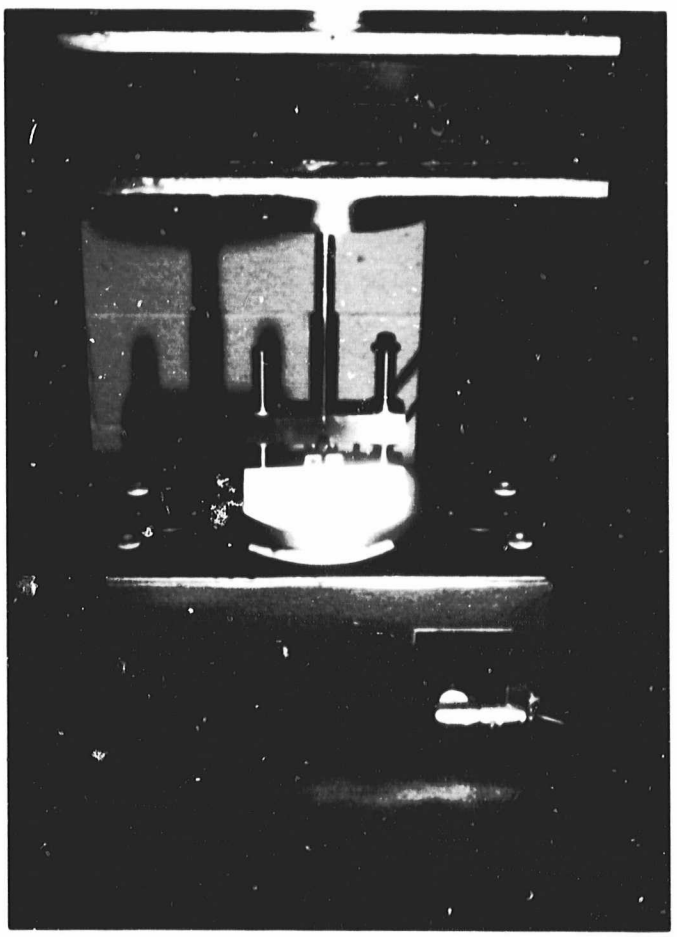


Figure 3. b. Picture of a three-point bending tester, when attached to an Instron.

of this experiment are given in Section 3.5 according to ASTM STP 521.[32]

3.3 Procedures

3.3.1 Electron Irradiation

3.3.1.1 Preconditioning

The specimens were placed in a heated vacuum desiccator at 80°C for 7 days in order to pre dry the specimens. Then the specimens were placed side by side on aluminum foil (Reynolds Wrap, heavy duty thickness 0.025 - 0.030 mm) and the ends of the specimens secured in place with a thin layer of scotch tape (see Figure 4). The aluminum foil was folded and the edges were sealed with an epoxy glue (Devcon 5-minute Epoxy®). An open glass tube was inserted prior to sealing the foil to permit a vacuum line to be connected for further vacuum treatment. The packages were then placed in the heated vacuum desiccator at 80°C for at least an additional 4 days. The glass tube was attached to a vacuum line and heat-sealed immediately prior to irradiation.

3.3.1.2 Electron Irradiation Exposure

The sealed packages were taken immediately after finishing the preconditioning to the electron accelerator and exposed to the radiation. Each package was placed in a Ziploc polyethylene bag (10" x 10") that was prefilled with nitrogen gas; the bag then was connected to a conveyor belt and passed through the electron accelerator. Each revolution of the conveyor through the beam resulted in a 10 Mrad dosage.

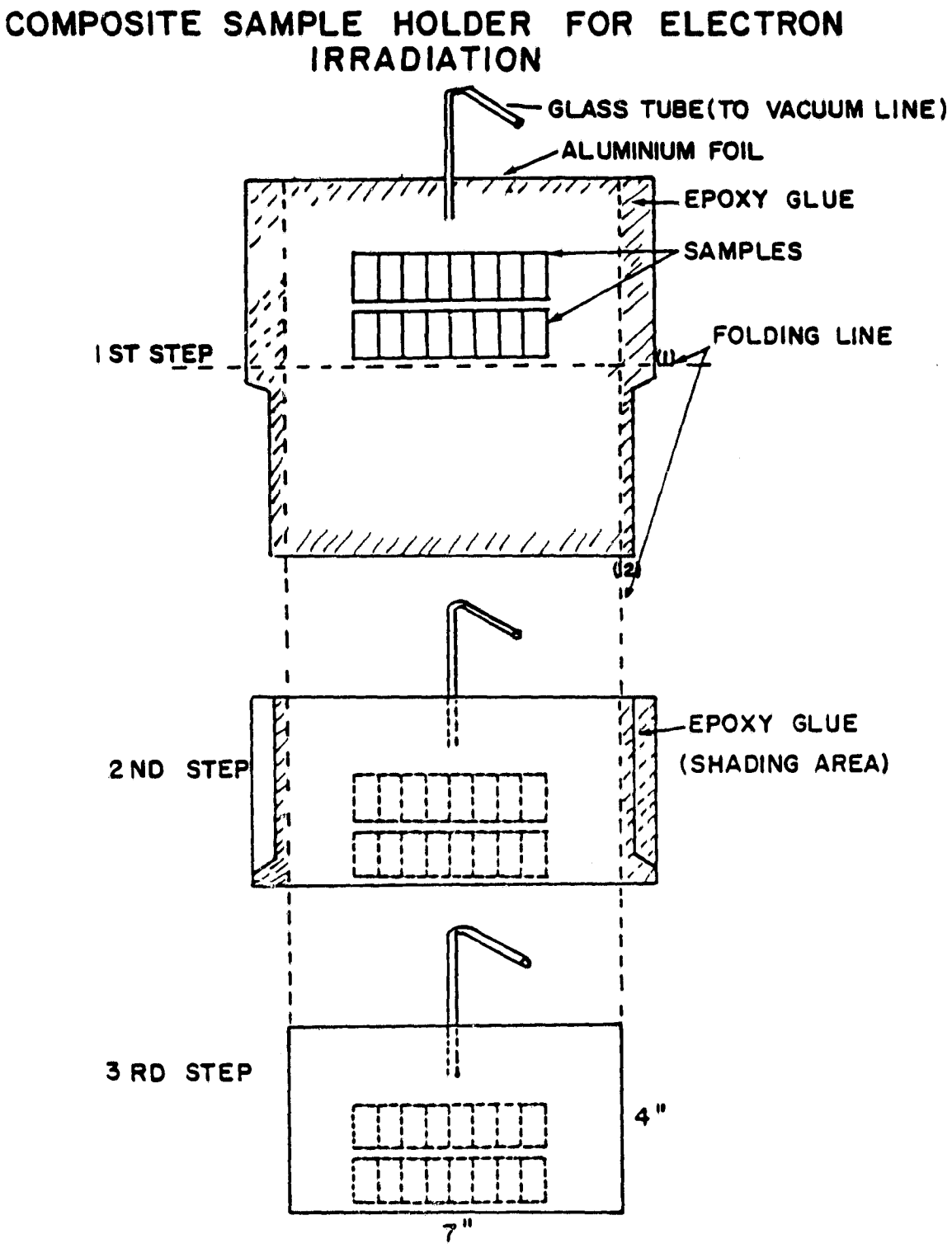


Figure 4. Preparation of sample holder for electron accelerator experiments.

3.3.1.3 Post Conditioning or Standard Conditioning

After the electron irradiation exposure (or the gamma irradiation exposure), the specimens were removed from the packages and placed in a standarized room at 20°C, 65% relative humidity, where they remained for about 2 weeks prior to mechanical testing.

3.3.2 Gamma Irradiation

The specimens were vacuum desiccated at 80°C for a minimum of 3 days, then placed in the vacuum chamber of the gamma cell, vacuum treated for 24 hours and exposed to gamma radiation for varying periods of time at a rate ≈ 0.33 Mrad/hr. Then the samples were conditioned in the standardize room as described in section 3.3.1.3.

3.4 Mechanical Testing

The mechanical tests, longitudinal flexual strength were conducted on an Instron machine, using a "three-point bending tester" attachment (see Figure 3b), according to ASTM D790[30] and ASTM STP 674.[31] The specimens were tested at a constant rate of elongation perpendicular to the plane of the composite at a speed of 0.254 cm/min. A span length of 1.40 cm was used.

The mechanical bending curves (load and deflection) were recorded on chart paper and the ultimate stress and the average modulus were calculated by using the standard equations for small bending deformation of elastic bodies as shown below:[30]

$$\text{Ultimate stress} = \frac{\text{Breaking load} \times \text{Spanlength} \times 1.5}{\text{Width} \times (\text{Thickess})^2} \qquad (1)$$

$$\text{Average modulus} = \frac{\text{Ultimate stress} \times (\text{Spanlength})^2}{6 \times \text{Thickness} \times \text{Deflection}} \qquad (2)$$

3.5 Tensile Test of Graphite Filament

Dry bundle test was used to test the graphite filaments.[32] Three-inch (75 mm) segments of the graphite bundle were weighed to obtain yarn denier, then placed across the center of 1-inch gauge length tab as shown in Figure 5 and secured to the tab edge with masking tape. The fils in the twisted yarn (i.e., T300) are of uniform length and tension as is; however, slight manual pretensioning and removal of loose fils from two segments are required to insure uniform loading. The bundles were then fastened to the tab using epoxy glue to prevent slippage in the grip. The tension test was carried out by gripping the tab in the jaws and loading the specimens to break at a 2% per minute strain rate (0.02 in/min for 1 in. gauge length). The breaking strength and the average modulus were calculated in kilogram per (centimeter)^2 (kg/cm^2). Then the strength values of the unirradiated and irradiated samples were compared.

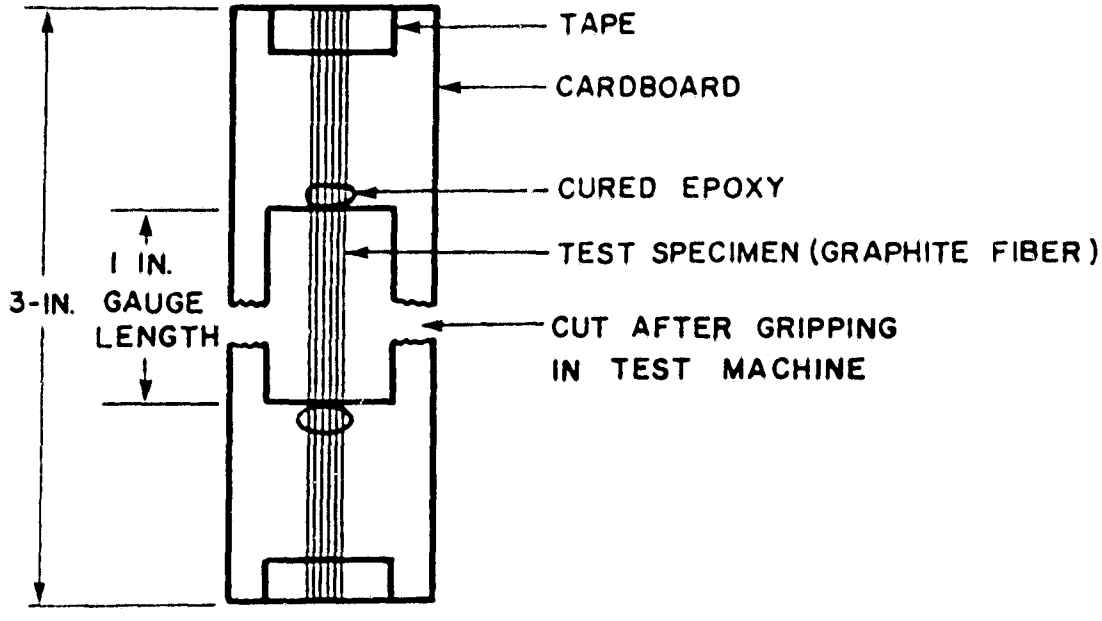


Figure 5. Tab mounted dry graphite bundle tension test specimen.

4. Results and Discussion

An empirical estimate of irradiation dosage as a function of penetration depth into unit density material is shown in Figure 6.[33] The characteristics of this curve is in good agreement with the experimental curve (Figure 7) which was obtained from irradiation of the radiachromic films (with incorporated aminotriphenyl methane dye derivatives made by Far West Technology Company) inside aluminum foil packages of varying thickness. These films become colored on radiation. The calibration curve of this coloration, as shown in Figure 8, was obtained using the Gamma Cell 220, Cobalt-60 source with known dose rate of 0.25 Mrad per hour. Since the electron accelerator was designed to pass samples across the beam twice during each revolution of the conveyor (once on the front side of the sample and once on the back side), the approximate dosage experienced by the composite specimens as a function of penetration depth is shown in Figure 9. The density of the composite is assumed to be about 1.55 gm/cm^3. The effective radiation dosage in the center of the specimen is approximately 39% higher than the edges.

The load and deformation curves of the composites under the three-point bending tests were approximately linear in all cases, as shown in Figure 10. The deflections were small, i.e., 1.5 - 1.7% so that equations 1 and 2 (Section 3.4) give excellent approximation of ultimate stress and average modulus.

The effects of electron and gamma radiations on the ultimate stress and the average modulus of the composites will be reported first for each type of composite studied, then they will be discussed together

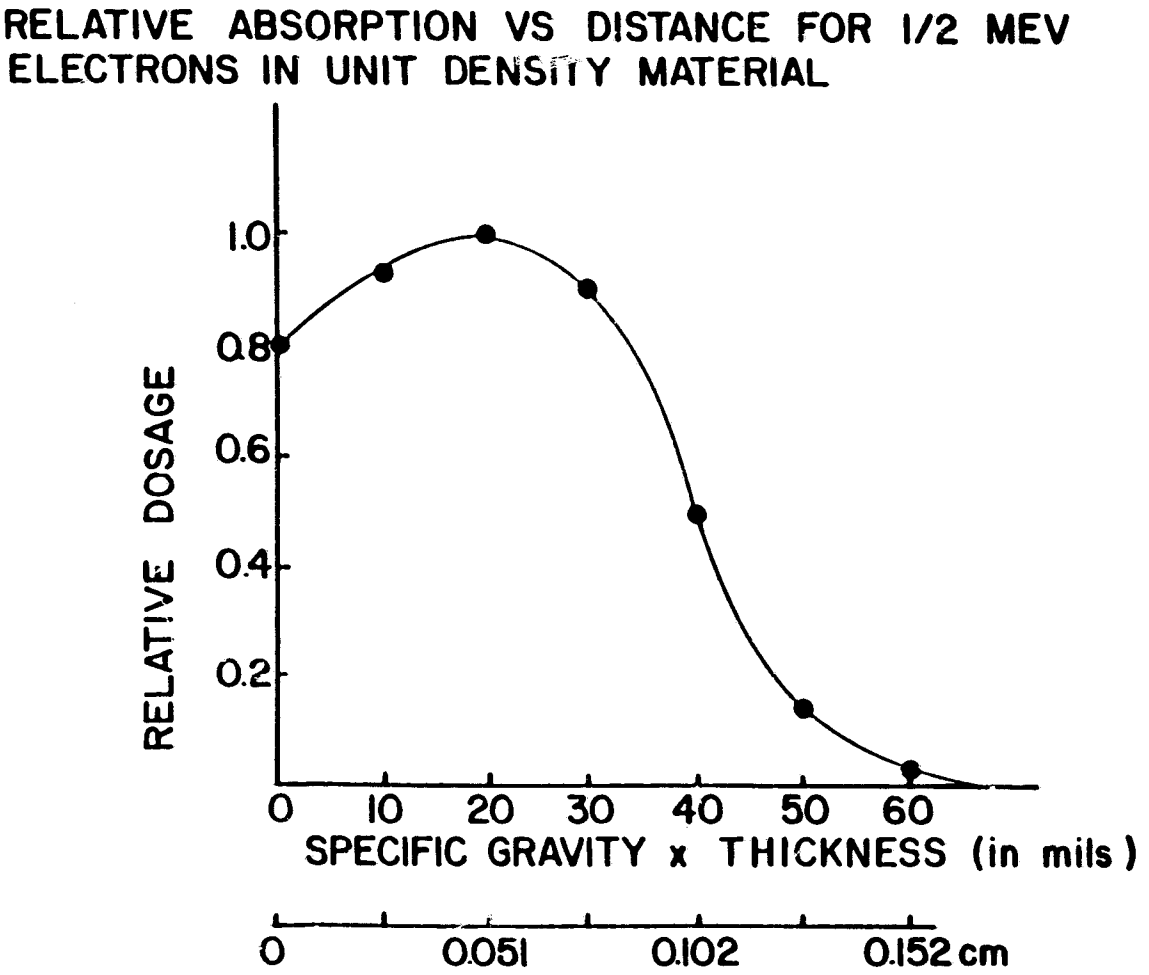


Figure 6. Sample dosage vs thickness of sample for 0.5 MeV electron Radiation (Theory).

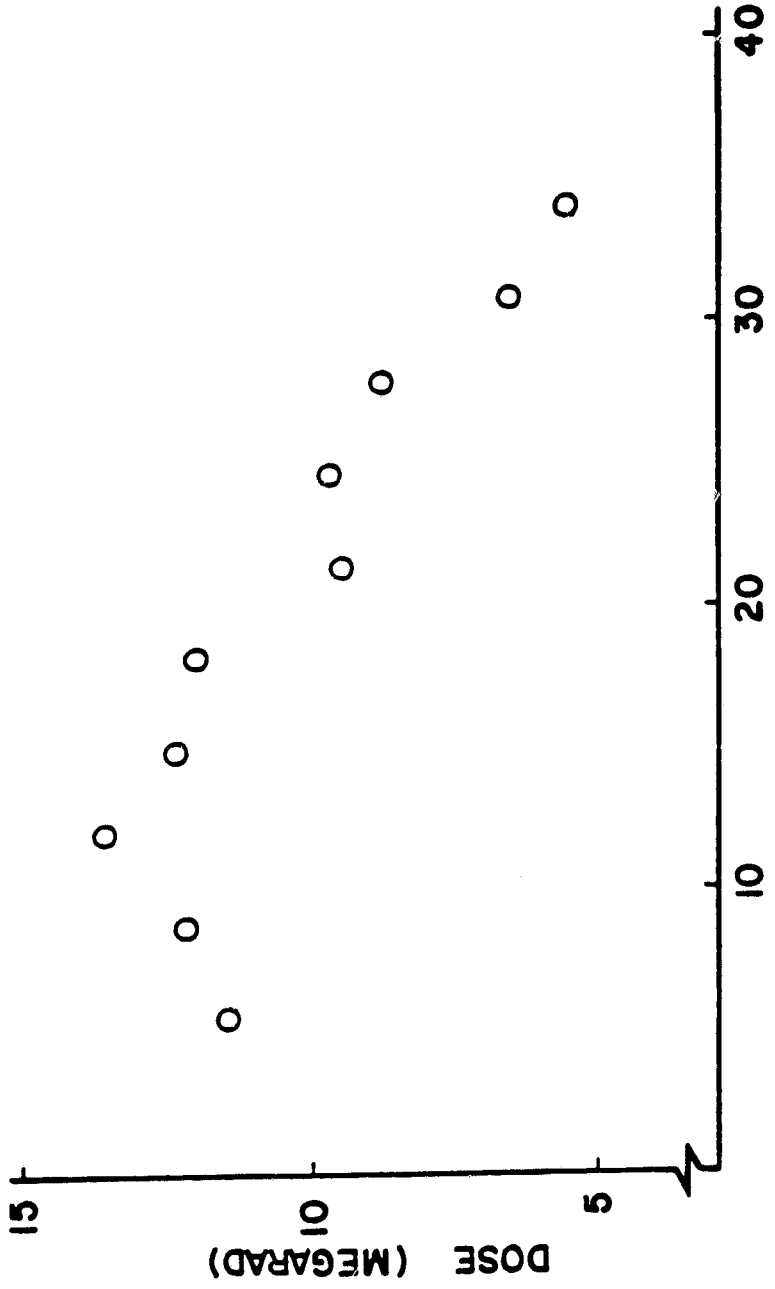


Figure 7. Sample Dosage vs thickness of sample for 0.5 MeV electron Radiation (experiment).

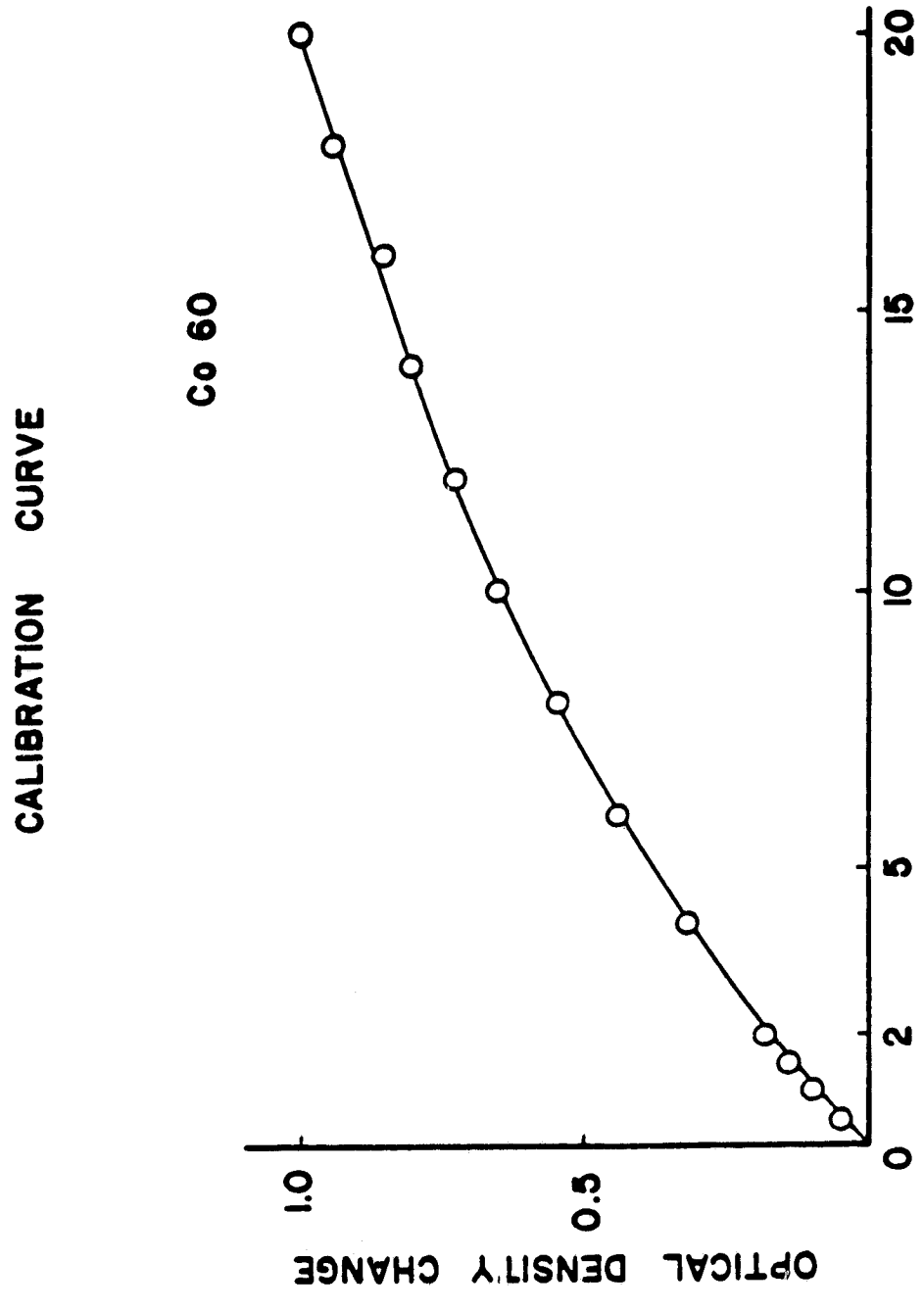


Figure 8. Calibration Curve: Dose (from Gamma cell) vs Optical density change.

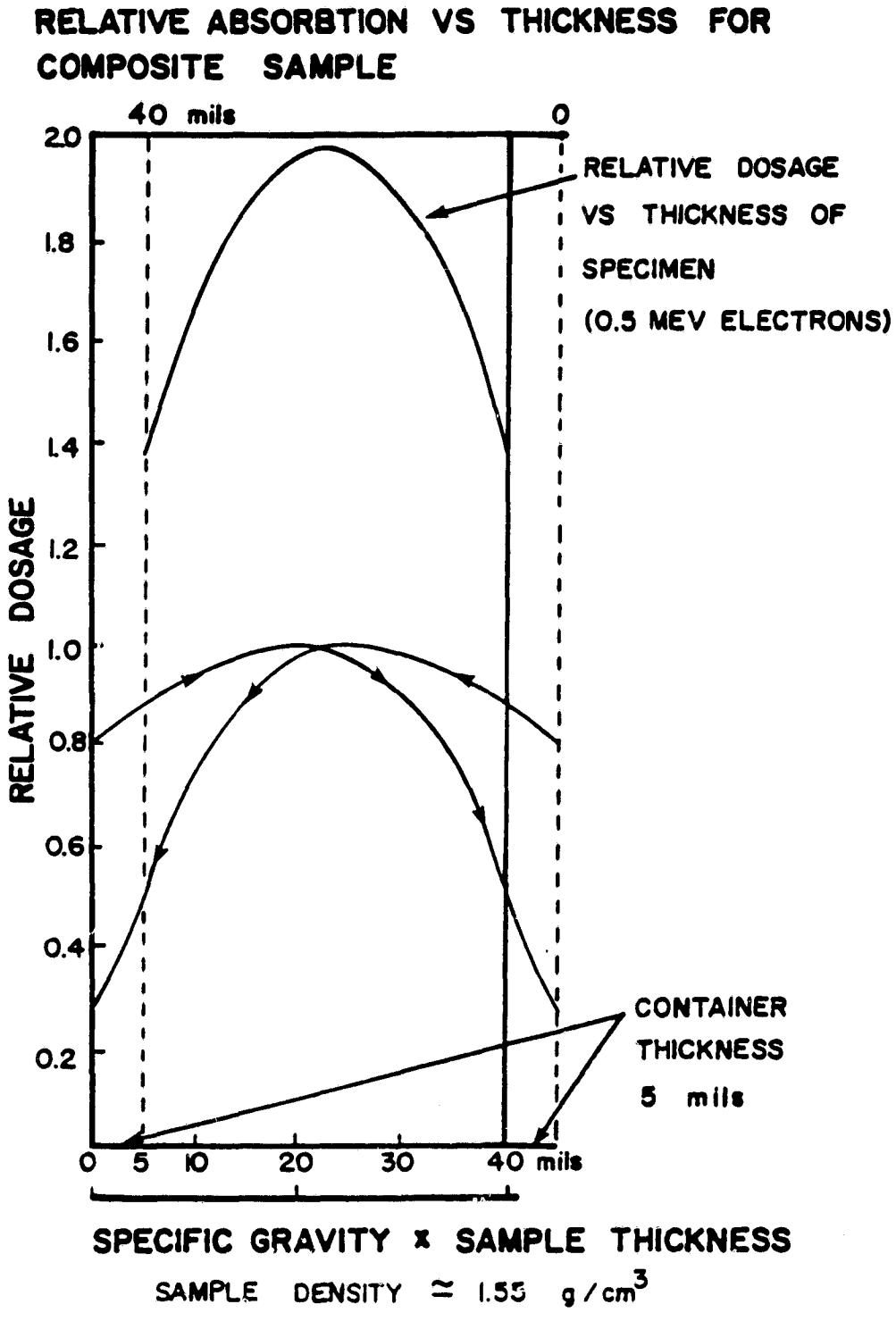


Figure 9. Relative absorption vs thickness for composite sample.

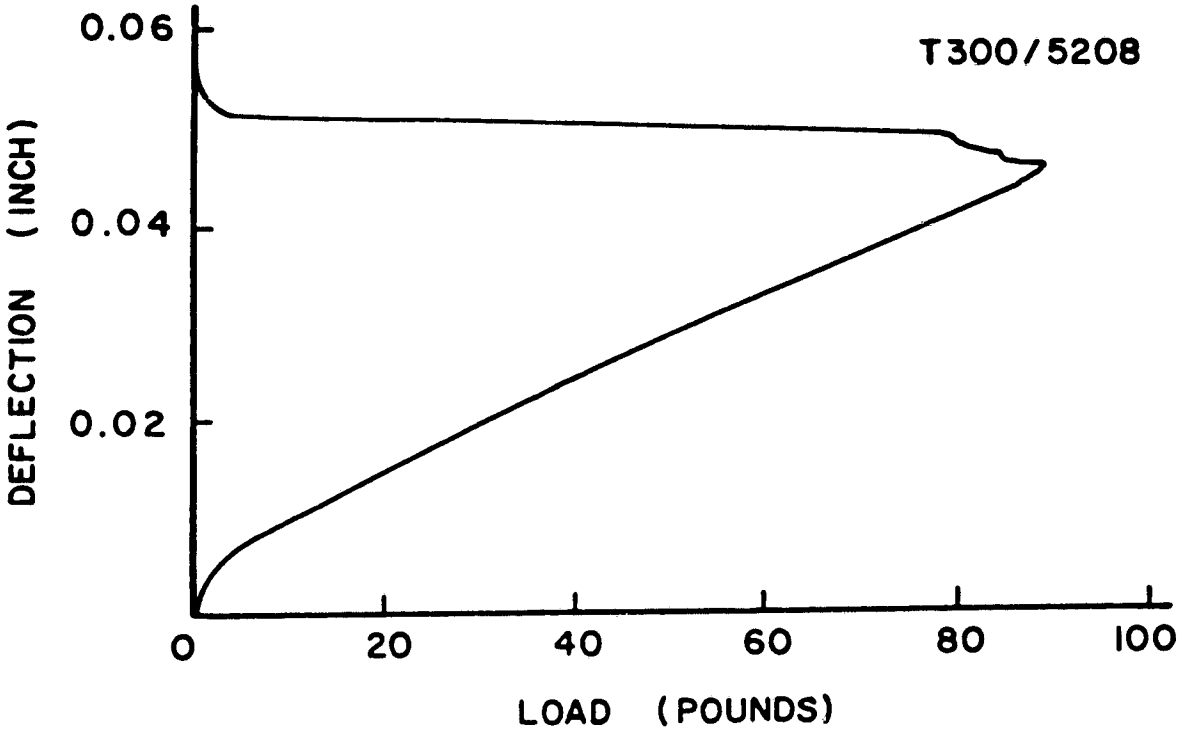


Figure 10. Load-Deflection Curve of the graphite reinforced composite from the Instron.

since the response of all composites to the radiation treatments is about the same.

4.1 T300/5208

4.1.1 Electron Irradiation

Two sets of T300/5208 (graphite/epoxy) samples, fabricated at different times, were irradiated by 0.5 Mev electron radiation at two different times using the same experimental conditions. The first set was irradiated up to 800 Mrad; the second set was irradiated up to 5000 Mrad. The mechanical responses of the first set are reported in Tables 1-2 and Figures 11-12.

Table 1 shows the details of the control treatment which is composed of at least 10 test specimens as replicates. The width and thickness of each specimen were measured by a vernier micrometer, significant up to 0.01 mm. The breaking loads and the breaking deflections were obtained from the graphs recorded by the Instron during the tests. The ultimate stress and average modulus were calculated using equations 1 and 2 (Section 3.4) by computer and statistically analyzed using SAS (Statistical Analysis System).[34] The average value of stress and modulus, the standard deviations of these parameters and the coefficients of variation for each exposure treatment are reported in Table 2.

Figures 11 and 12 show the breaking stress and the average modulus plotted versus electron dose, respectively. The 95% confident intervals of the true means were calculated as shown below:

$$\text{Upper limit} = \bar{X} + t_{(.95,\ n-1)}\frac{s}{\sqrt{n}}$$

Table 1. Mechanical Properties of Sample and T300/5208 Control (heat 80°C in Vacuum as Precondition but no Radiation Treatment)

[redacted]	Thickness (cm)	Width (cm)	Breaking load (kg.)	Strain*	Ultimate Stress (kg/cm^2)	Average Modulus (kg/cm^2)
1	0.056	1.276	40.27	0.017	21,090	1,269,000
2	0.054	1.279	36.82	0.016	20,690	1,257,000
3	0.057	1.278	39.09	0.017	19,730	1,136,000
4	0.053	1.278	36.64	0.015	21,380	1,374,000
5	0.055	1.276	39.09	0.015	21,220	1,373,000
6	0.055	1.275	39.54	0.016	21,490	1,352,000
7	0.054	1.272	40.27	0.017	27,750	1,349,000
8	0.056	1.270	42.73	0.017	22,480	1,318,000
9	0.057	1.272	38.18	0.015	19,360	1,242,000
10	0.056	1.271	39.09	0.015	20,550	1,358,000
11	0.057	1.270	40.91	0.016	20,770	1,297,000
Average					21,050	1,302,000
Standard Deviation					1,017	72,500
C.V.%					4.83	5.56

*Maximum strain [30] = $\frac{6\,Dd}{L^2}$

D is maximum deflection, cm
d is thickness of specimen, cm
L is support span = 1.397 cm

Table 2. Mechanical Properties of Sample Set T300/5208 (Set A) as Function of Electron Irradiation Dosage

[redacted]	Heat in vac., no radiation	10 Mrad	50 Mrad	100 Mrad	200 Mrad	400 Mrad	800 Mrad
Pre-cond.* (days)	8	8	8	8	8	8	8
Post-cond.** (days)	56	56	56	56	56	56	56
No. of specimen	11	11	11	11	11	11	11
Ult. stress (kg/cm^2)	21050	21350	21550	22130	22870	22080	21830
Standard Deviation	1017	913	1297	793	1179	970	861
C.V.%	4.83	4.28	6.02	3.58	7.78	4.39	3.94
Analysis of Variance *** 5%	A	A	A	AB	B	AB	AB
Analysis of Variance *** 10%	A	AB	AB	BC	C	BC	AB
Average modulus (kg/cm^2)	1302000	1322000	1344000	1389085	1436000	1338000	1337000
Standard Deviation	72500	60700	95700	59900	171400	58500	51[illegible]00
C.V.%	5.56	4.59	7.12	4.31	11.93	4.37	3.82
Analysis of Variance *** 5%	A	A	A	AB	B	A	A
Analysis of Variance *** 10%	A	AB	AB	BC	C	AB	AB

***Duncan's Multiple Range Analysis — Means with the same letter are not significantly different. The polynomial regression analysis in more informative for explaining mechanical dose response effects than the Duncan analysis (See Table 3)

** Post Treatment — 65% R.H., 20°C.

* Pretreatment — 80°C in vacuum desiccator.

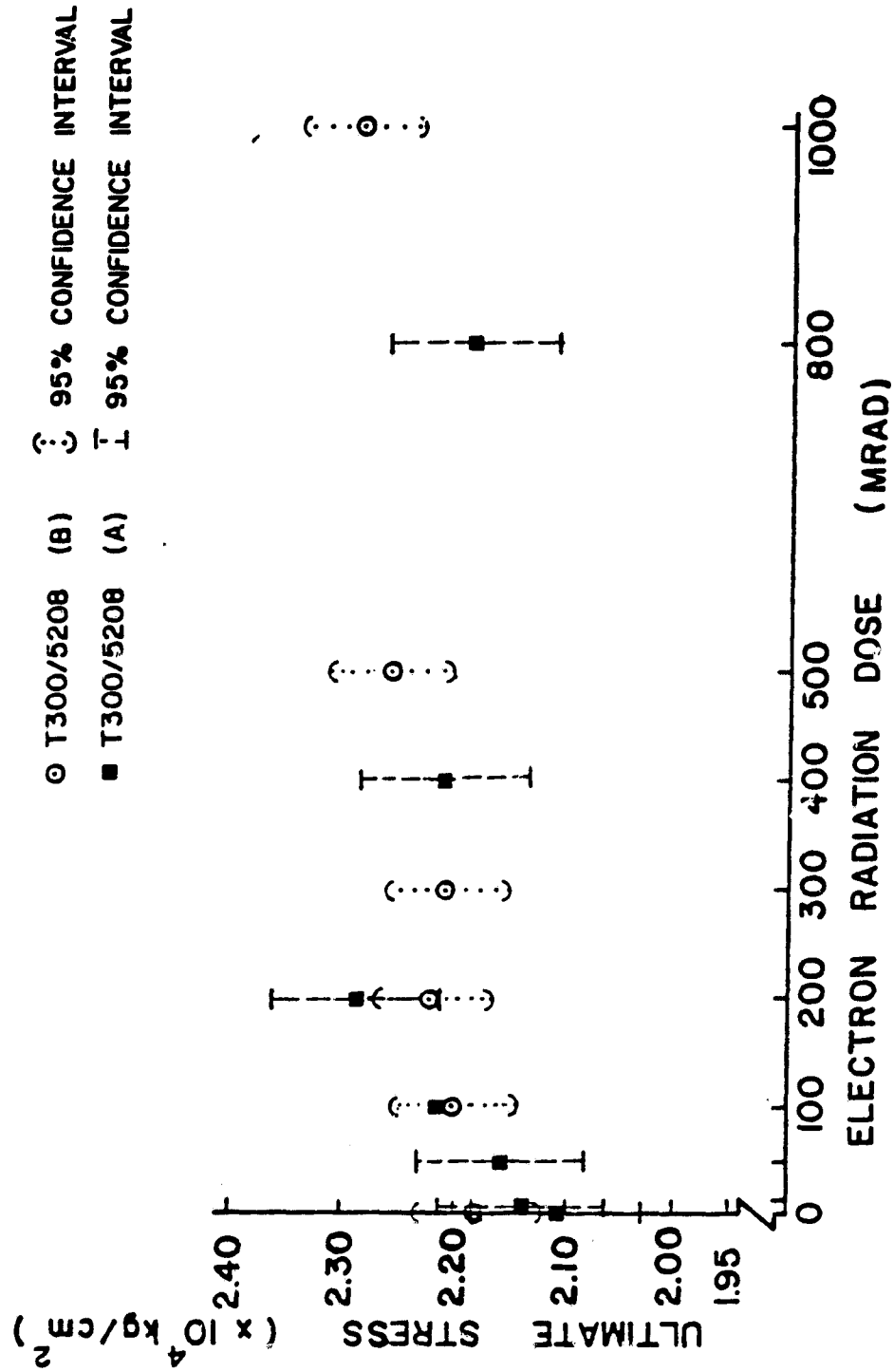


Figure 11. Ultimate stress of T300/5208 (set A and set B) vs electron dose (0-1000 Mrad).

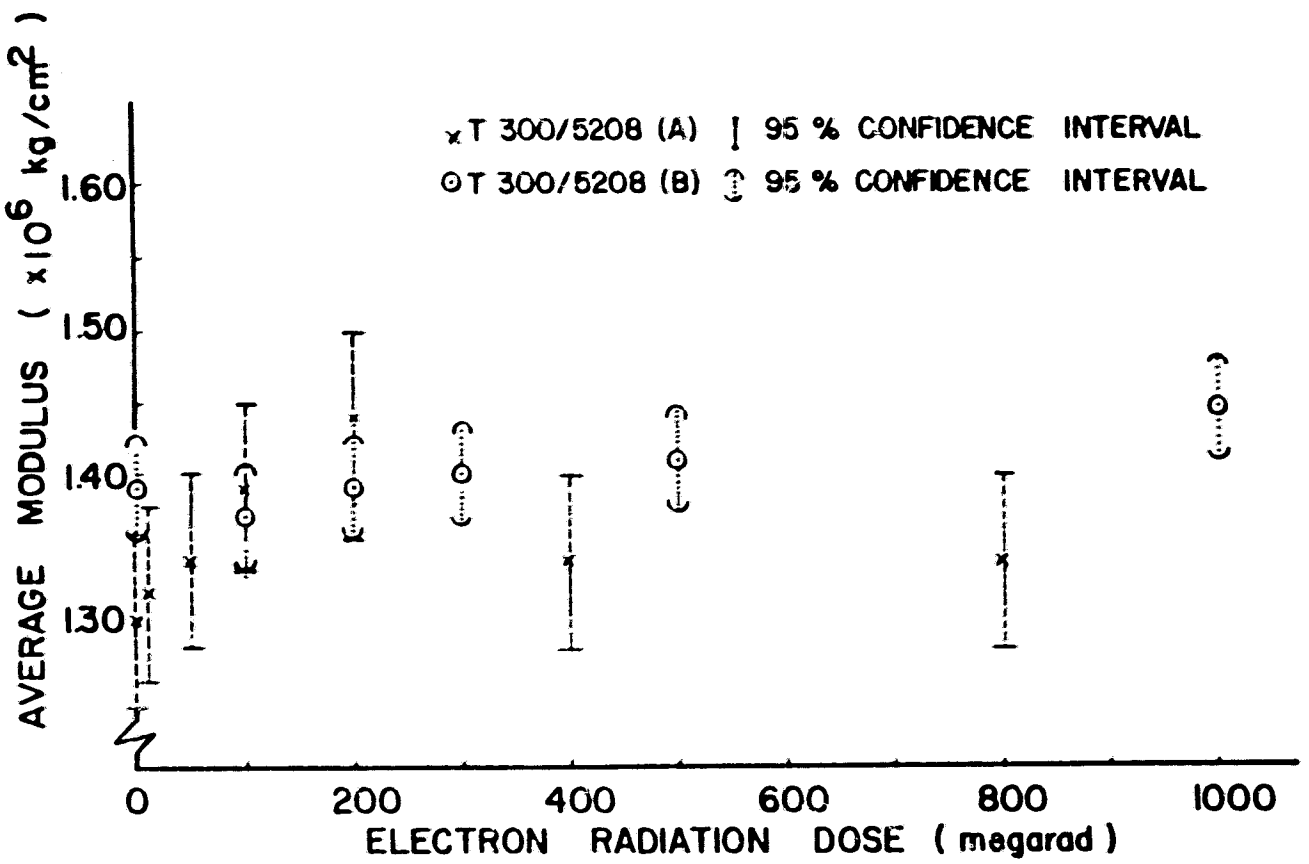


Figure 12. Average Modulus of T300/5208 (set A and set B) vs electron dose (0-1000 Mrad).

$$\text{Lower limit} = \bar{X} - t_{(.95,\ n-1)}\frac{s}{\sqrt{n}}$$

$\bar{X}$ is the average value for each exposure treatment (i.e., the average value of 11 specimens in Table 1),

s is the pooled standard deviation of one experimental set

n is the number of the test specimens for each exposure treatment (i.e., n = 11 in Table 2), and

t is the statistical value obtained from the student's t-distribution.

All irradiated samples show a slight increase in stress and modulus compared with the control. At 200 Mrad, the stress and modulus are at maxima value and are 9% and 10% higher than the control values, respectively. The effects of electron radiation were analyzed using the Duncan's multiple range analysis and polynomial regression analysis. The means were compared at the 5% and 10% significant level using the Duncan's multiple range analysis. (They are not significantly different if they are characterized by the same letter (Table 2).) From this test, the maximum values are significantly higher than the control values.

The polynomial regression analysis was computed by using SAS and PROC GLM (General Linear Model). The quadratic regression equations fit the data for the effect of electron dose on the stress and modulus of the first set of composites at 5% significane level.

$Y = 21,340 + 5.97X - 0.0068X^2$ for stress

$Y = 1,331,000 + 320.51X - 0.4060X^2$ for modulus

Y is the dependent variable, e.g., stress or modulus.

X is the independent variable, e.g., dose in Mrad.

Table 3 summarizes the value and significance level of all parameters in all the regression equations for every type of sample used in this study.

These data suggest that the effect of electron radiation for sample set A up to 800 Mrad results in a significant increase in the stress and modulus of this graphite/epoxy up to maxima (at 200 Mrad), then the stress and modulus decrease. However, the stress value at 800 Mrad is 3.7% higher than the control value.

In order to study the behavior of T300/5208 at higher electron dose levels, the second set of T300/5208 was exposed up to 5000 Mrad. The mechanical responses are shown in Table 4 and Figures 13-14. The calculations were done the same way as before (in Table 2 and Figures 11-12). The samples show a small monotonic increase in stress and modulus compared with the control. At 5000 Mrad, the stress and the modulus are 13% and 11% higher, respectively. These are significantly higher than the control values.

For the effect of electron radiation up to 5000 Mrad on stress and modulus of T300/5208 samples used in this study, a cubic regression equation fit the data at the 5% significance level for the stress and modulus, respectively. The equations are shown below; the values and significance level of all the parameters are also compared in Table 3.

Table 3. Values and the Significance Level of the Coefficient of the Regression Equations: $y = a + bx^2 + cx^2 + dx^3$*

Sample Identification	Expose Type & Maximum Dosage	Coefficient of Regression Equation							
		a		b		c		d	
		Value (kg/cm^2)	% Significance Level	Value ($\frac{kg/cm^2}{Mrad}$)	% Significance Level	Value ($\frac{kg/cm^2}{(Mrad)^2}$)	% Significance Level	Value ($\frac{kg/cm^2}{(Mrad)^3}$)	% Significance Level
T300/ 5208									
Stress	Electron	21,340	1	5.97	1	-0.0068	1	-	-
Modulus	800 Mrad	1,331,000	1	320.51	5	-0.4060	5	-	-
Stress	Electron 5000 Mrad	21,790	1	1.61	1	-0.0006	1	7.4×10^{-8}	1
Modulus		1,375,000	1	96.34	1	-0.0343	5	4.3×10^{-6}	5
Stress	Gamma	21,100	1	18.74	5	-0.1154	5	-	-
Modulus	167 Mrad	1,287,000	1	2897.47	1	-17.5379	1	-	-
AS/ 3501-6									
Stress	Gamma	13,950	1	2.73	5	-	-	-	-
Modulus	357 Mrad	1,051,000	1	138.73	10	-	-	-	-
C6000/ PMR15									
Stress	Electron	20,230	1	0.83	1	-	-	-	-
Modulus	800 Mrad	1,110,000	1	89.24	1	-	-	-	-
Stress	Electron 5000 Mrad	21,160	1	1.27	5	-0.0007	5	1.0×10^{-7}	1
Modulus		1,033,000	1	117.02	1	-0.0656	1	9.3×10^{-6}	1
C6000/ P1700									
Stress	Electron	20,300	1	0.38	1	-	-	-	-
Modulus	5000 Mrad	1,238,000	1	24.17	1	-	-	-	-

*y is the predicted value of stress or modulus and x is the irradiation dose in Mrad.

Table 4. Mechanical Properties of Sample Set T300/5208 (Set B) as Function of Electron Irradiation Dosage

[redacted]	Heat in vac., no radiation	100 Mrad	200 Mrad	300 Mrad	500 Mrad	1000 Mrad	2000 Mrad	3000 Mrad	4000 Mrad	5000 Mrad
Pre-cond. (days)*	11	11	11	11	11	11	11	11	11	11
Post-cond. (days)**	21	21	21	21	21	21	21	21	21	21
No. of specimen	10	10	10	10	10	10	10	10	10	10
Ult. stress (Kg/cm^2)	21780	21970	22180	22060	22570	22870	23240	23540	23630	24670
Standard Deviation	706	790	719	735	862	767	569	713	914	837
C.V.%	3.24	3.60	3.24	3.30	3.82	3.36	2.45	3.03	3.87	3.39
Analysis of Variance *** 5%	G	FG	EFG	FG	DEF	CDE	BCD	BC	B	A
Analysis of Variance *** 10%	F	EF	EF	EF	DE	CD	BC	B	B	A

Average modulus (kg/cm^2)		1389000	1374000	1392000	1401000	1409000	1447000	1473000	1462000	1495000	154000
Standard Deviation		44900	57600	65900	48800	54000	59600	23800	48400	52000	44200
C.V.%		3.23	4.20	4.73	3.48	3.84	4.12	1.62	3.31	3.48	2.87
Analysis of Variance ***	5%	D	D	D	CD	CD	BC	B	B	AB	A
Analysis of Variance ***	10%	D	D	D	D	D	C	BC	BC	B	A

***Duncan's Multiple Range Analysis — Means with the same letter are not significantly different. The polynomial regression analysis is more informative for explaining mechanical dose response effects than the Duncan analysis. (See Table 3)

**Post Treatment — 65% R.H., 20°C.

*Pre Treatment — 80°C in vacuum desiccator.

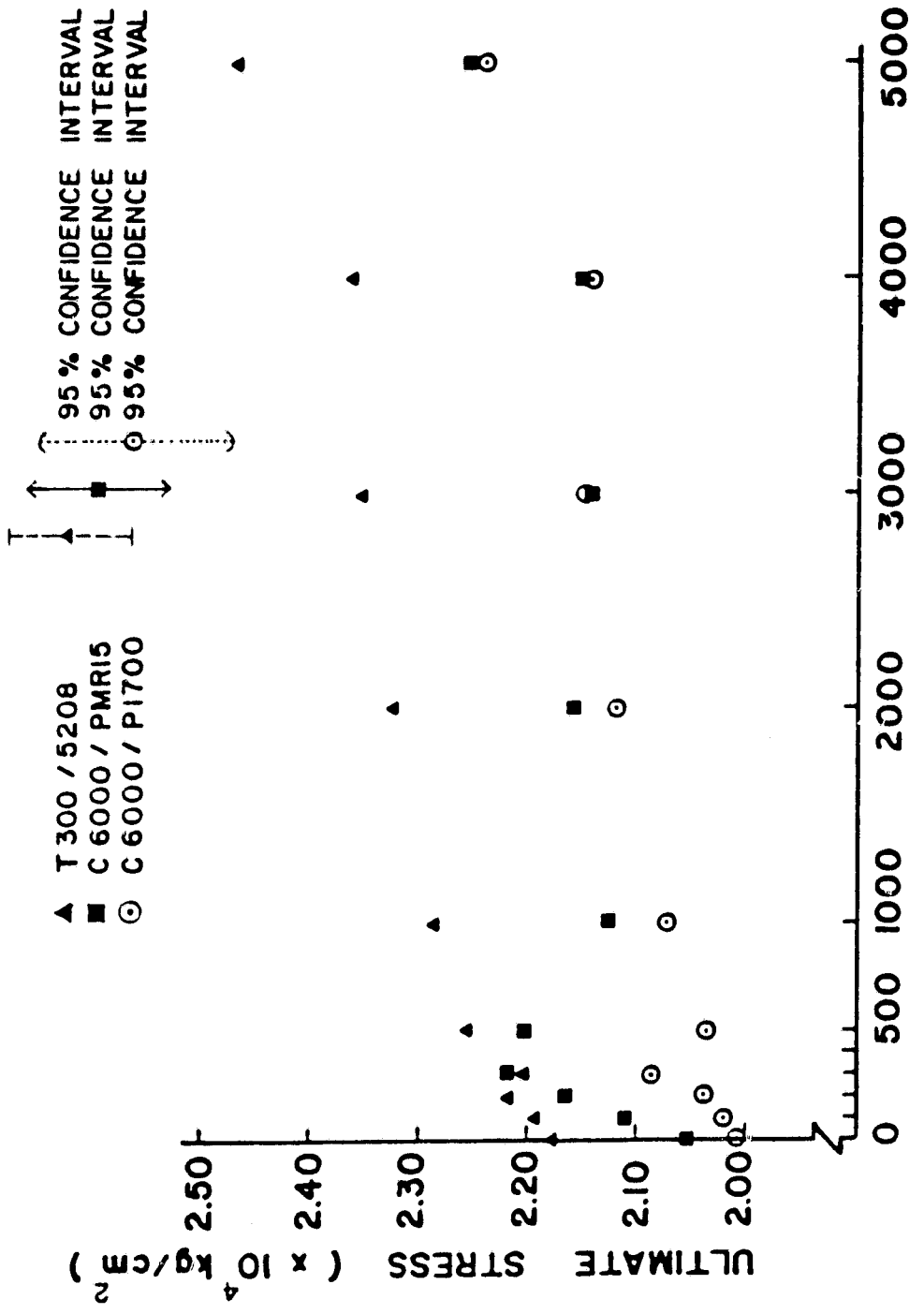


Figure 13. Ultimate Stress of T300/5208 (set B), C6000/PMR15 (set B) and C6000/ P1700 vs electron dose (0-5000 Mrad).

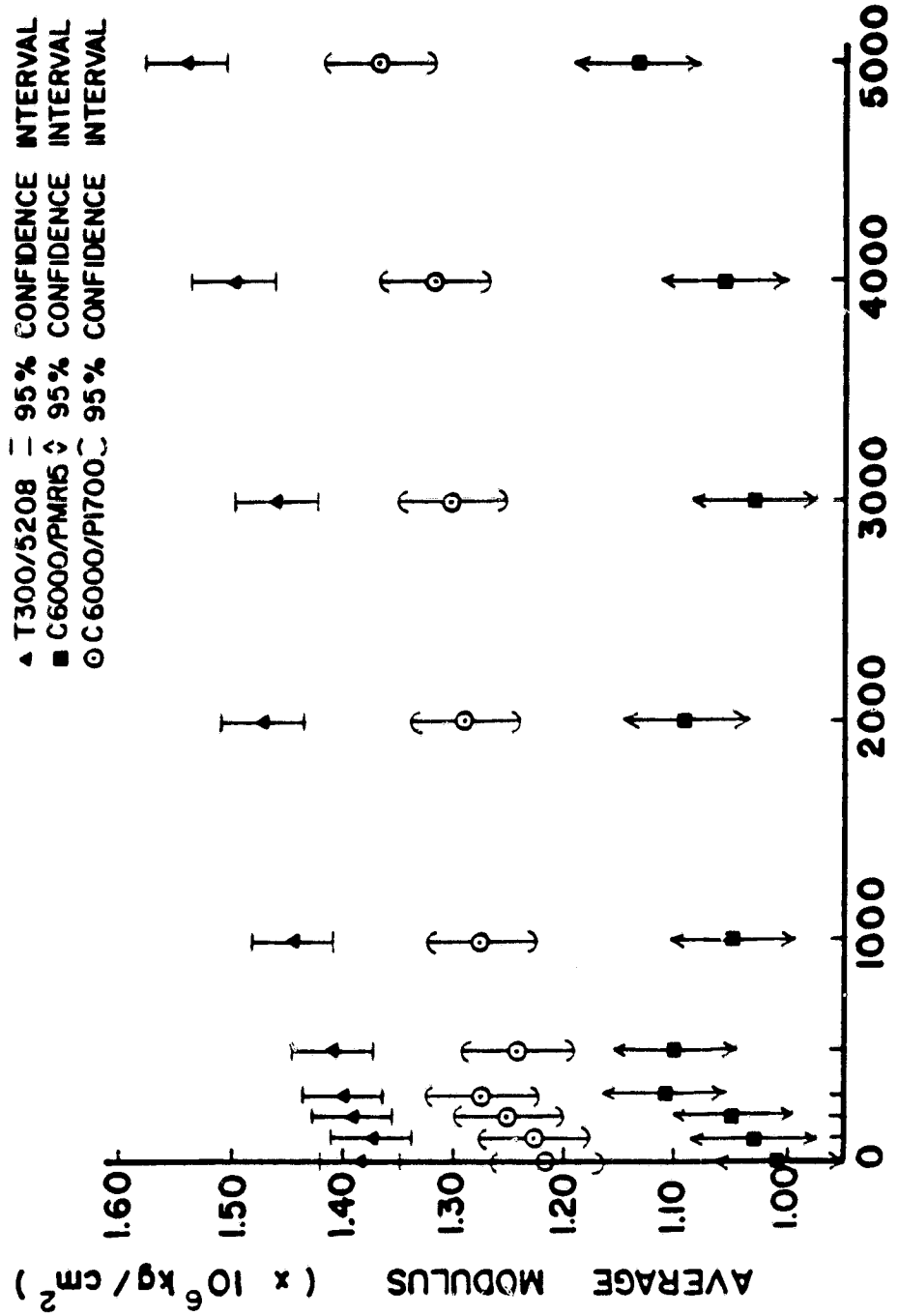


Figure 14. Average Modulus of T300/5208 (set B), C6000/PMR-15 (set B) and C6000/P1700 vs electron dose (0-5000 Mrad).

$Y = 21{,}790 + 1.61X - 0.0006X^2 + (7.4 \times 10^{-8})X^3$ for stress

$Y = 1{,}375{,}000 + 96.34X - 0.0343X^2 + (4.3 \times 10^{-6})X^3$ for modulus

The data suggests there is an increase in strength and modulus with electron dose up to 5000 Mrad.

The results of sample set A and set B (just for the first 1000 Mrad) are compared in the same plot in Figures 11 and 12. Sample set B shows a monotonic increase in strength up to 1000 Mrad while sample set A shows a maximum at 200 Mrad (which may not be a true maximum, due to experimental error). The differences in these two sets may be due to time variations in fabrication (e.g., curing condition), in irradiation (e.g., voltage variation of the current beam), in heating and vacuum conditions, or in the testing conditions. However, from the effect of electron radiation up to 5000 Mrad, it can be safely concluded that the strength and modulus for the T300/5208 samples used in this study do not degrade rapidly under high energy radiation in an inert environment.

4.1.2 Gamma Radiation

The T300/5208 sample used in this study was fabricated at the same time as set A and was irradiated using 1.33 Mev gamma radiation at a dose rate of approximately 0.33 Mrad per hour for periods up to 500 hours. Ten to fifteen test specimens were removed from the Gamma Cell, after 124, 250, 500 exposure hours, which resulted in the gamma dose of approximately 41, 83, 167 Mrad, respectively. All the samples were kept in standard conditions atleast 2 weeks before testing them at the same time. The strength and modulus of the irradiated samples were compared with the unirradiated samples in Table 5. The plots of ultimate stress

Table 5. Mechanical Properties of Sample Set T300/5208 (Set A) as a Function of Exposure to Gamma Irradiation Dosage (Dose rate ca. 1/3 Mrad/hr)

[redacted]		Control	41 Mrad	83 Mrad	167 Mrad
Pre-cond. (days)*		7	3	3	3
Post-cond. (days)**		72	73	73	73
No. of specimen		10	13	13	13
Ultimate stress (Kg/cm^2)		21120	21650	21890	21010
Standard Deviation		826	853	836	1152
C.V.%		3.91	3.94	3.82	5.98
Analysis of Variance ***	5%	AB	AB	A	B
	10%	B	AB	A	B
Average modulus (kg/cm^2)		1279000	1391000	1395000	1284000
Standard Deviation		76100	54100	55500	106900
C.V.%		5.95	3.89	3.99	8.33
Analysis of Variance ***	5%	B	A	A	B
	10%	B	A	A	B

***Duncan's Multiple Range Analysis — Means with the same letter are not significantly different. The polynomial regression analysis is more informative for explaining mechanical dose response effects than the Duncan analysis. (See Table 3)

**Post Treatment — 65% R.H., 20°C.

*Pre Treatment — 80°C in vacuum desiccator.

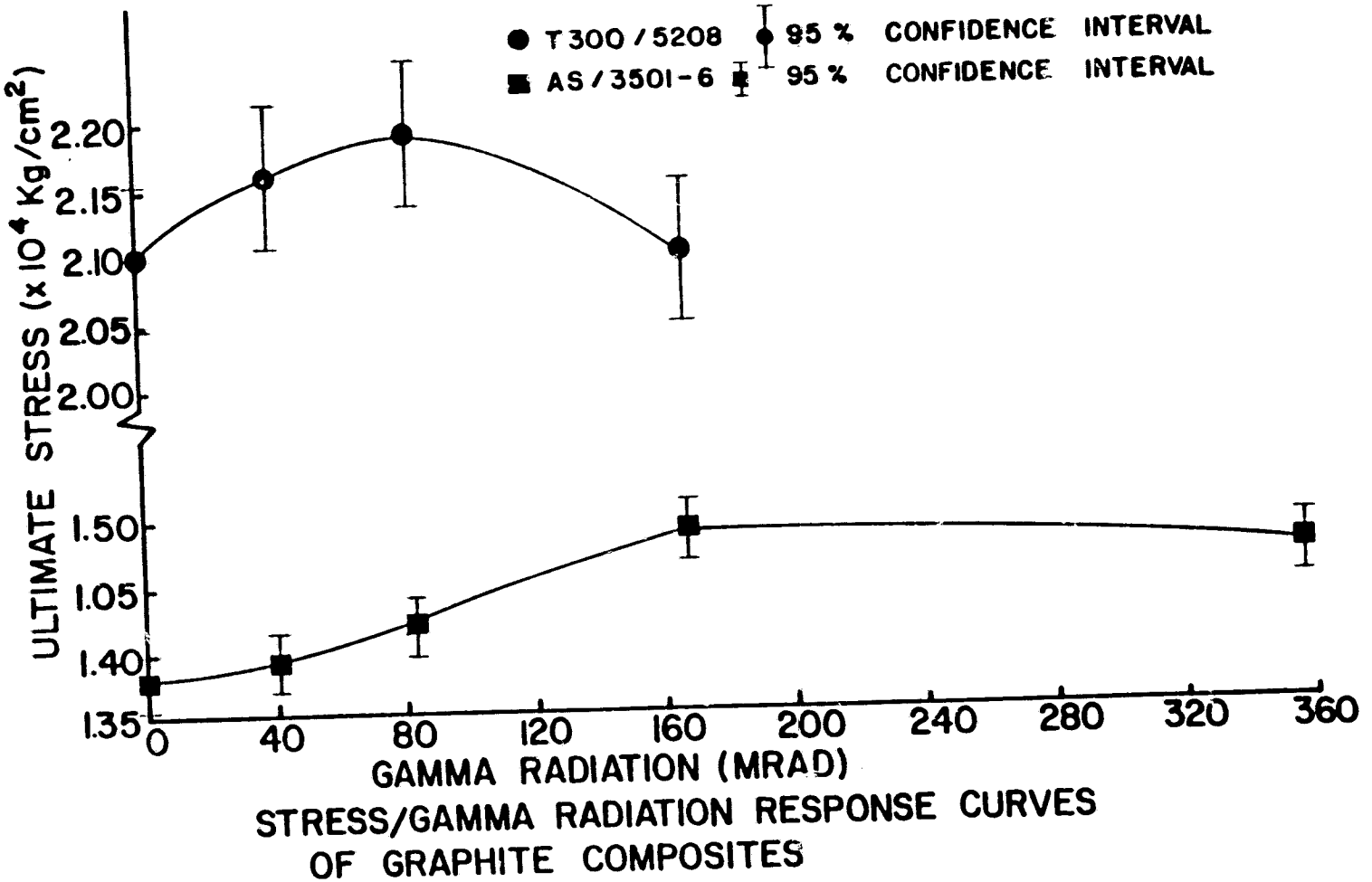


Figure 15. Ultimate stress of T300/5208 (set A) and AS/3501-6 vs gamma dose (0-357 Mrad).

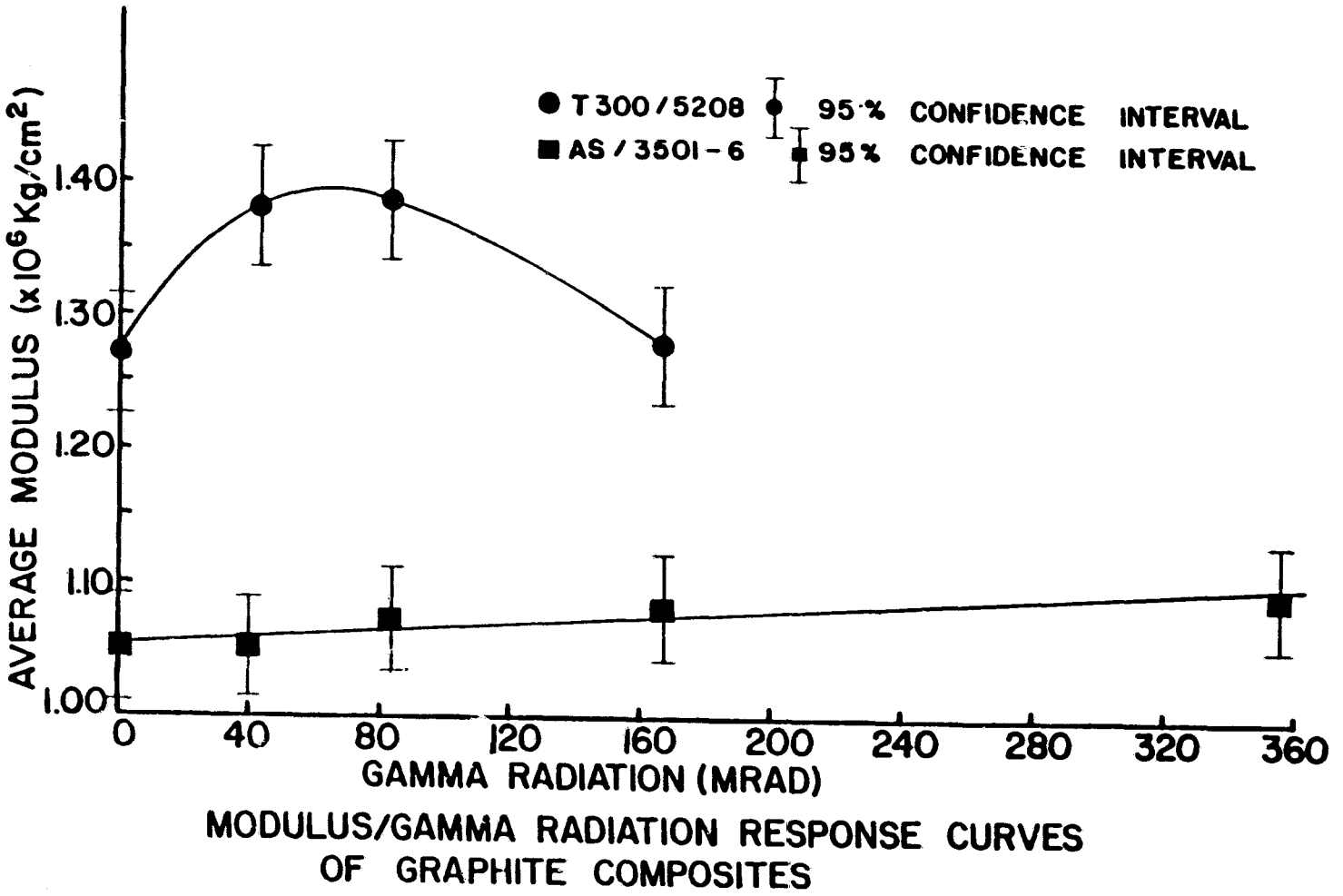


Figure 16. Average Modulus of T300/5208 (set A) and AS/3501-6 vs gamma dose (0-357 Mrad).

and average modulus versus doses are shown in Figures 15-16. The maximum value of stress occurred at 83 Mrad, is 4% higher than the control value, and the difference is not significant at the 5% level (using Duncan's multiple range analysis). The maximum value of modulus occurred at 41 and 83 Mrad, is about 9% higher than the control value, and the difference is significant at the 5% level. At 167 Mrad, the stress and modulus decreased from the value at 83 Mrad, but was about the same as the control value.

The polynomial regression equations fit the mechanical responses of composites to gamma radiation dose up to 167 Mrad at 5% significance level, as shown:

$$Y = 21,100 + 18.74X - 0.11X^2 \quad \text{for stress}$$

and $Y = 1,287,000 + 2897.46X - 17.54X^2$ for modulus

The identification of X and Y is the same as described before (Section 4.1.1). The data suggests that the gamma radiation up to 167 Mrad causes a small increase in the ultimate stress and the average modulus of T300/5208 up to the maxima (at 83 Mrad). Then the stress and modulus decrease to about the same as the control value. However, much larger exposures are needed in order to determine if the trend of decreasing strength and modulus beyond 167 Mrad continues.

4.2 AS/3501-6

Gamma Irradiation

One set of AS/3501-6 graphite/epoxy samples were irradiated using 1.33 Mev gamma radiation at a known dose rate of approximately 0.33 Mrad per hour for varying periods of time, using the same

conditions as described in Section 4.1.2. The gamma doses were approximately 41, 83, 167 and 357 Mrad. The mechanical responses are reported in Table 6 and Figures 15-16. The results show an approximately monotonic increase in the stress and modulus as radiation dose increases.

The maximum stress occurred at 167 Mrad and was 8% higher than the control value. Table 6 shows the Duncan's multiple range comparison. The samples exposed to 167 Mrad have significantly higher stress and modulus values than the control. The stress of the samples exposed to 367 Mrads was slightly less than that at 167 Mrad. This can be represented by a linear polynomial regression equation:

$Y = 13,950 + 2.73X$ for stress at 5% significance level

which suggests that a gamma dose up to 357 Mrad linearly increases the ultimate stress of the AS/3501-6 composite.

For the average modulus, the maximum value is at 357 Mrad and is 5% higher than the control value. This difference is not significant at the 5% level. A linear regression equation:

$Y = 1,051,000 + 138.73X$ fit the data at 10% significance level.

This AS/3501-6, graphite/epoxy composite showed similar behavior to T300/5208 (Set B) when exposed to electron radiation. For AS/3501-6, the stress and modulus increases approximately linearly with the exposure dose over the range of radiation treatments that we studied. At this stage, we do not know the effect of further gamma radiation.

Table 6. Mechanical Properties of Sample Set AS/3501-6 as a Function of Exposure to Gamma Radiation (Dose rate ca. 1/3 Mrad/hr)

[redacted]	Heat in vac., no radiation	40 Mrad	83 Mrad	167 Mrad	357 Mrad
Pre-cond. (days)*	7	3	3	3	3
Post-cond. (days)**	78	78	78	78	78
No. of specimen	20	20	20	20	20
Ult. stress (Kg/cm^2)	13810	13900	14210	14910	14700
Standard Deviation	1207	1970	1539	1574	1398
C.V.%	8.74	14.18	10.83	10.56	9.51
Analysis of Variance *** 5%	B	AB	AB	A	AB
Analysis of Variance *** 10%	B	B	AB	A	AB
Average modulus (kg/cm^2)	1047000	1051000	1066000	1083000	1095000
Standard Deviation	49200	103500	97400	91610	103800
C.V.%	4.68	9.85	9.13	8.46	9.64
Analysis of Variance *** 5%	A	A	A	A	A
Analysis of Variance *** 10%	A	A	A	A	A

***Duncan's Multiple Range Analysis — Means with the same letter are not significantly different. The polynomial regression analysis is more informative for explaining mechanical dose response effects than the Duncan analysis. (See Table 3)

**Post Treatment — 65% R.H., 20°C.

*Pre Treatment — 80°C in vacuum desiccator.

4.3 C6000/PMR15

Electron Irradiation

Two sets of C6000/PMR-15, graphite/polyimide samples (set A and set B), fabricated at different times, were irradiated by 0.5 Mev electron acceleration at two different times, but under the same experimental conditions. The first set was irradiated up to 800 Mrad, and the second set was irradiated up to 5000 Mrad. The mechanical responses of the first set were reported in Table 7 and Figures 17 and 18.

Figures 17 and 18 show a continuous increase in the stress and modulus with the electron dose. For set A at 800 Mrad, the stress is 3% higher than the control value. This increased value is not significant at the 5% level, but is significant at the 10% level. The maximum modulus occurred at 800 Mrad and is 7% higher than the control value. This difference is significant at the 5% significance level.

Linear polynomial regression equations: $Y = 20,230 + 0.83X$ and $Y = 1,110,000 + 89.24X$ fit the data at the 1% significance level for the effect of electron dose on the stress and modulus, respectively. X and Y are defined in the same way as in Section 4.1.1. These two equations suggest that the effect of electron dose is to linearly increase the stress and modulus.

The ultimate stress and the average modulus of the second set irradiated up to 5000 Mrad are reported in Table 8 and Figures 13 and 14. At 5000 Mrad, the ultimate stress and the average modulus were maxima and were 10% and 12% higher than the control values, respectively. A cubic regression equation fits the data at 5% significance

Table 7. Mechanical Properties of Sample Set C6000/EMR-15 (Set A) as a Function of Electron Irradiation Dosage

[redacted]	Heat in vac., no radiation	10 Mrad	50 Mrad	100 Mrad	200 Mrad	400 Mrad	800 Mrad
Pre-cond.* (days)	8	8	8	8	8	8	8
Post-cond.** (days)	35	35	35	35	35	35	31
No. of specimen	13	13	13	13	13	13	13
Ult. stress (kg/cm^2)	20180	20090	20240	20380	20600	20630	20810
Standard Deviation	849	771	867	536	751	466	748
C.V.%	4.21	3.84	4.28	2.63	3.64	2.26	3.59
Analysis of Variance *** 5%	AB	A	AB	AB	AB	AB	B
Analysis of Variance *** 10%	AB	A	AB	ABC	ABC	BC	C

Average modulus (kg/cm^2)	1094000	1103000	1114000	1130000	1149000	1145000	1175000
Standard Deviation	45600	67200	68402	39910	51610	39940	49200
C.V.%	4.17	6.09	6.14	3.53	4.49	3.49	4.18
Analysis of Variance *** 5%	A	AB	ABC	ABC	CD	BCD	D
Analysis of Variance *** 10%	A	A	AB	AB	BC	BC	C

***Duncan's Multiple Range Analysis — Means with the same letter are not significantly different. The polynomial regression analysis is more informative for explaining mechanical dose response effects than the Duncan analysis. (See Table 3)

** Post Treatment — 65% R.H., 20°C.

* Pretreatment — 80°C in vacuum desiccator.

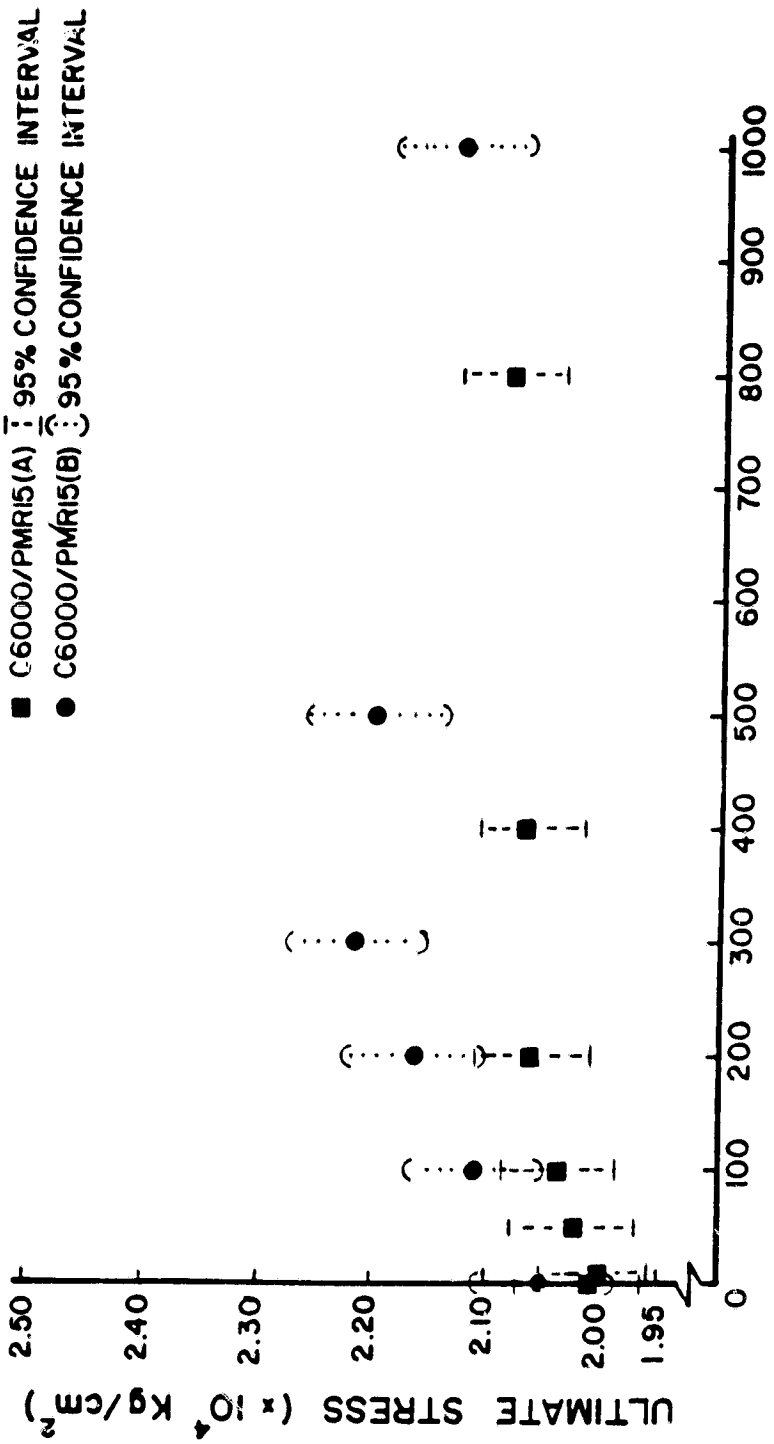


Figure 17. Ultimate stress of C6000/PMR-15 (set A and set B) vs electron dose (0-1000 Mrad).

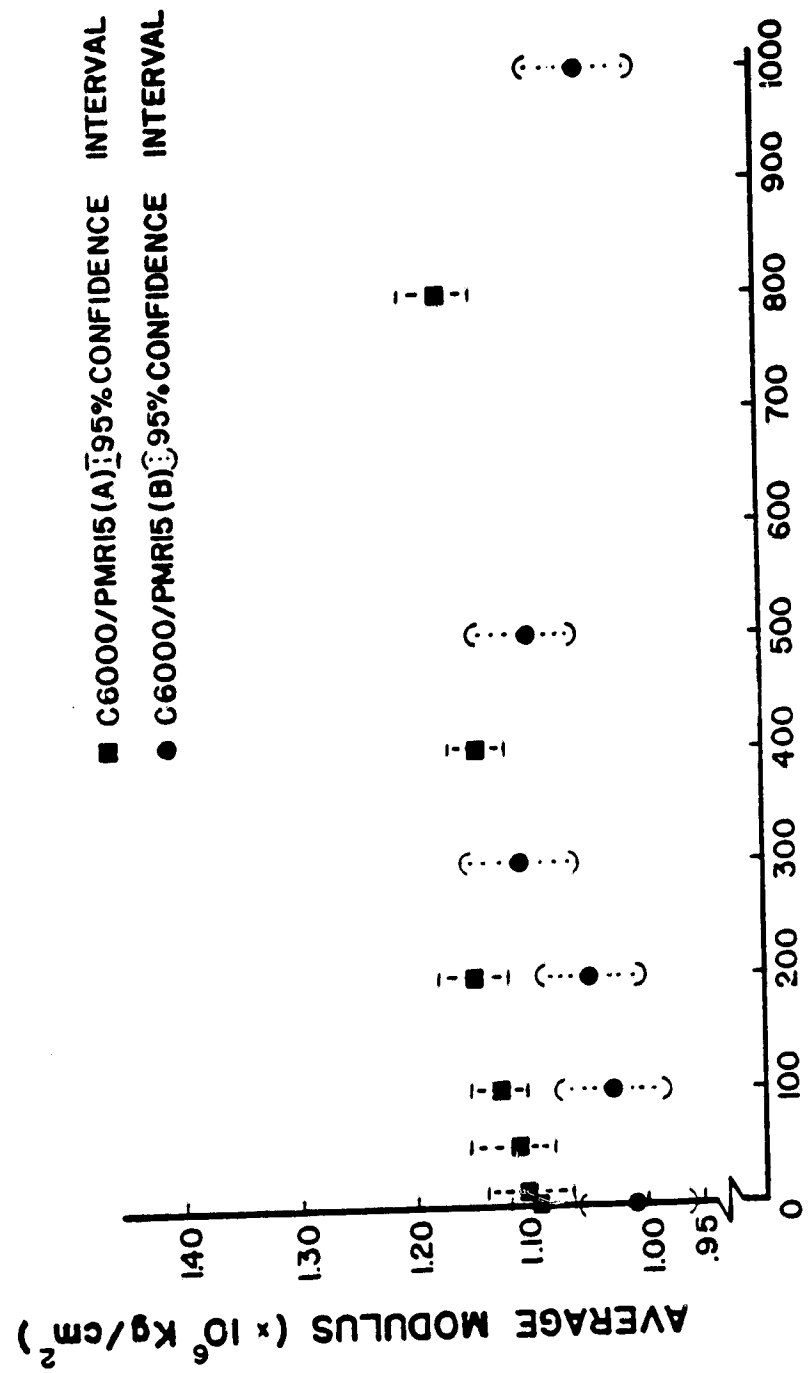


Figure 18. Average Modulus of C6000/PMR-15 (set A and set B) vs electron dose (0-1000 Mrad).

Table 8. Mechanical Properties of Sample Set C5000/BMR15 (Set B) as a Function of Electron Irradiation Dosage

[redacted]	Heat in vac., no radiation	100 Mrad	200 Mrad	300 Mrad	500 Mrad	1000 Mrad	2000 Mrad	3000 Mrad	4000 Mrad	5000 Mrad
Pre-cond. (days)*	11	11	11	11	11	11	11	11	11	11
Post-cond. (days)**	24	24	24	24	24	24	24	24	24	24
No. of specimen	11	11	11	11	11	11	11	11	11	11
Ult. stress (Kg/cm^2)	20530	21080	21670	22160	22020	21250	21590	21440	21500	22560
Standard Deviation	500	783	908	800	971	647	1077	1886	827	705
C.V.%	2.44	3.71	4.19	3.61	4.41	3.05	4.99	8.80	3.85	3.12
Analysis of Variance *** 5%	D	CD	ABC	AB	ABC	BCD	BC	BC	BC	A
Analysis of Variance *** 10%	E	DE	BCD	AB	ABC	CDE	BCD	BCD	BCD	A

Average modulus (kg/cm^2)		1010000	1029000	1049000	1110000	1104000	1050000	1093000	1030000	1058000	1134000
Standard Deviation		36200	51400	67700	73900	67300	45900	111000	105400	63400	89000
C.V.%		3.58	4.99	6.45	6.66	6.10	4.37	10.15	10.23	5.99	7.84
Analysis of Variance ***	5%	D	CD	BCD	AB	AB	BCD	ABC	CD	BCD	A
	10%	C	C	BC	AB	AB	BC	AB	C	BC	A

***Duncan's Multiple Range Analysis — Means with the same letter are not significantly different. The polynomial regression analysis is more informative for explaining mechanical dose response effects the Duncan analysis. (See Table 3)

**Post Treatment — 65% R.H., 20°C.

*Pre Treatment — 80°C in vacuum desiccator.

level, for the effect of electron radiation up to 5000 Mrad on both the ultimate stress and the average modulus of C6000/PMR15 sample used in this study, as shown below:

$$Y = 21,160 + 1.27X - 0.0007X^2 + (1.0 \times 10^{-7})X^3 \text{ for stress}$$

$$Y = 1,033,000 + 117.02X - 0.0656X^2 + (19.3 \times 10^{-6})X^3 \text{ for modulus}$$

The results of sample set A and set B (just for the first 1000 Mrad) are compared in the same plot (Figures 17 and 18). Sample set A shows a linear increase in stress and modulus with the electron radiation up to 800 Mrad. Sample set B also shows an increase in stress and modulus with the electron radiation up to 5000 Mrad, which is in good agreement with set A.

4.4 C6000/P1700

Electron Irradiation

One set of C6000/P1700 graphite/polysulfone composites were irradiated with 0.5 Mev electron radiation up to 5000 Mrad. The mechanical responses were reported in Table 9 and Figures 13 and 14. The samples showed a slight increase in stress and modulus with increasing radiation doses. At 5000 Mrad, the stress and modulus were maxima and were 12% and 13% higher than the control values, respectively. By using the Duncan's multiple range analysis, these increases were significant.

Linear polynomial regression equations fit the data at the 1% significance level for the effect of electron radiation up to 5000 Mrad on the stress and modulus of C6000/P1700, as shown below:

$$Y = 20,300 + 0.38X \text{ for stress}$$

$$Y = 1,238,000 + 24.17X \text{ for modulus}$$

Table 9. Mechanical Properties of Sample Set C5000/P1700 as a Function of Electron Irradiation Dosage

[redacted]	Heat in vac., no radiation	100 Mrad	200 Mrad	300 Mrad	500 Mrad	1000 Mrad	2000 Mrad	3000 Mrad	4000 Mrad	5000 Mrad
Pre-cond. (days)*	11	11	11	11	11	11	11	11	11	11
Post-cond. (days)**	24	24	24	24	24	24	24	24	24	24
No. of specimen	10	10	10	10	10	10	10	10	10	10
Ult. stress (Kg/cm^2)	20080	20200	20380	20880	20350	20720	21180	21470	21440	22390
Standard Deviation	727	1106	924	1460	1145	740	903	1852	1934	947
C.V.%	3.62	5.48	4.53	6.99	5.63	3.57	4.26	8.62	9.02	4.23
Analysis of Variance *** 5%	C	BC	BC	BC	BC	BC	BC	AB	AB	A
Analysis of Variance *** 10%	D	CD	CD	BCD	CD	BCD	BC	B	B	A

Average modulus (kg/cm^2)		1218000	1228000	1249000	1274000	1241000	1277000	1292000	1302000	1318000	1372000
Standard Deviation		54600	55400	69500	134700	74200	53900	68000	58000	107800	85000
C.V.%		4.48	4.51	5.57	10.57	5.98	4.22	5.27	4.45	8.18	6.20
Analysis of Variance ***	5%	D	CD	BCD	BCD	BCD	BCD	BCD	ABC	AB	A
	10%	E	DE	CDE	BCDE	CDE	BCDE	BCD	BC	AB	A

***Duncan's Multiple Range Analysis — Means with the same letter are not significantly different. The polynomial regression analysis is more informative for explaining mechanical dose response effects than the Duncan analysis. (See Table 3)

**Post Treatment — 65% R.H., 20°C.

*Pre Treatment — 80°C in vacuum desiccator.

These equations suggest there is a linear increase in ultimate stress and average modulus of C6000/P1700 sample used in this study due to the effect of electron radiation dose up to 5000 Mrad.

4.5 T300 Graphite Fiber

Electron Irradiation

One set of T300 graphite filaments was irradiated with 0.5 Mev electron radiation after pre-vacuum treatment and without the presence of oxygen. The irradiation was up to 5000 Mrad. The tensile strength of these graphite filaments was determined by using the dry bundle test according to ASTM, STP 521.[32] The gauge length was 1-in. The crosshead speed of the Instron testing machine was 0.02 in/min. The tensile strengths and the average moduli of T300 after irradiation and the control value are reported in Table 10 and Figure 19.

The statistical analysis (Analysis of Variance) shows that the dose effect is not significant.

4.6 C6000 Graphite Fiber

Electron Irradiation

One set of C6000, graphite filaments, was irradiated with 0.5 Mev electron radiation using the same procedures described in the previous section. The tensile strengths and moduli of C6000 after irradiation and the control values are reported in Table 11 and Figure 20.

The statistical analysis (Analysis of Variance) shows that the dose effect is not sigificant.

4.7 Discussion

The experimental results of all types of graphite composites used in this study show that the effect of high energy radiation on the

Table 10. Tensile Strength of T300 as a Function of Electron Irradiation Dosage

[redacted]	Control	100 Mrad	200 Mrad	300 Mrad	500 Mrad	1000 Mrad	2000 Mrad	3000 Mrad	4000 Mrad	5000 Mrad
Pre-cond. (days)*	14	14	14	14	14	14	14	14	14	14
Post-cond. (days)**	105	105	105	105	105	105	105	105	105	105
No. of specimen	7	7	6	7	7	7	7	7	7	7
Tensile str. (Kg/cm^2)	6197	6088	6773	5917	7022	6306	7006	6166	6368	5963
Standard Deviation	867	911	444	708	888	304	999	972	1094	841
C.V.%	14.0	15.0	6.6	12.0	12.6	4.8	14.2	15.7	17.2	14.1
Average modulus (kg/cm^2)	283,700	308,800	338,600	300,100	313,100	300,300	322,700	345,300	356,600	350,800

*Pre Condition — 80°C in vacuum desiccator.
**Post Condition — 65% R.H., 20°C.

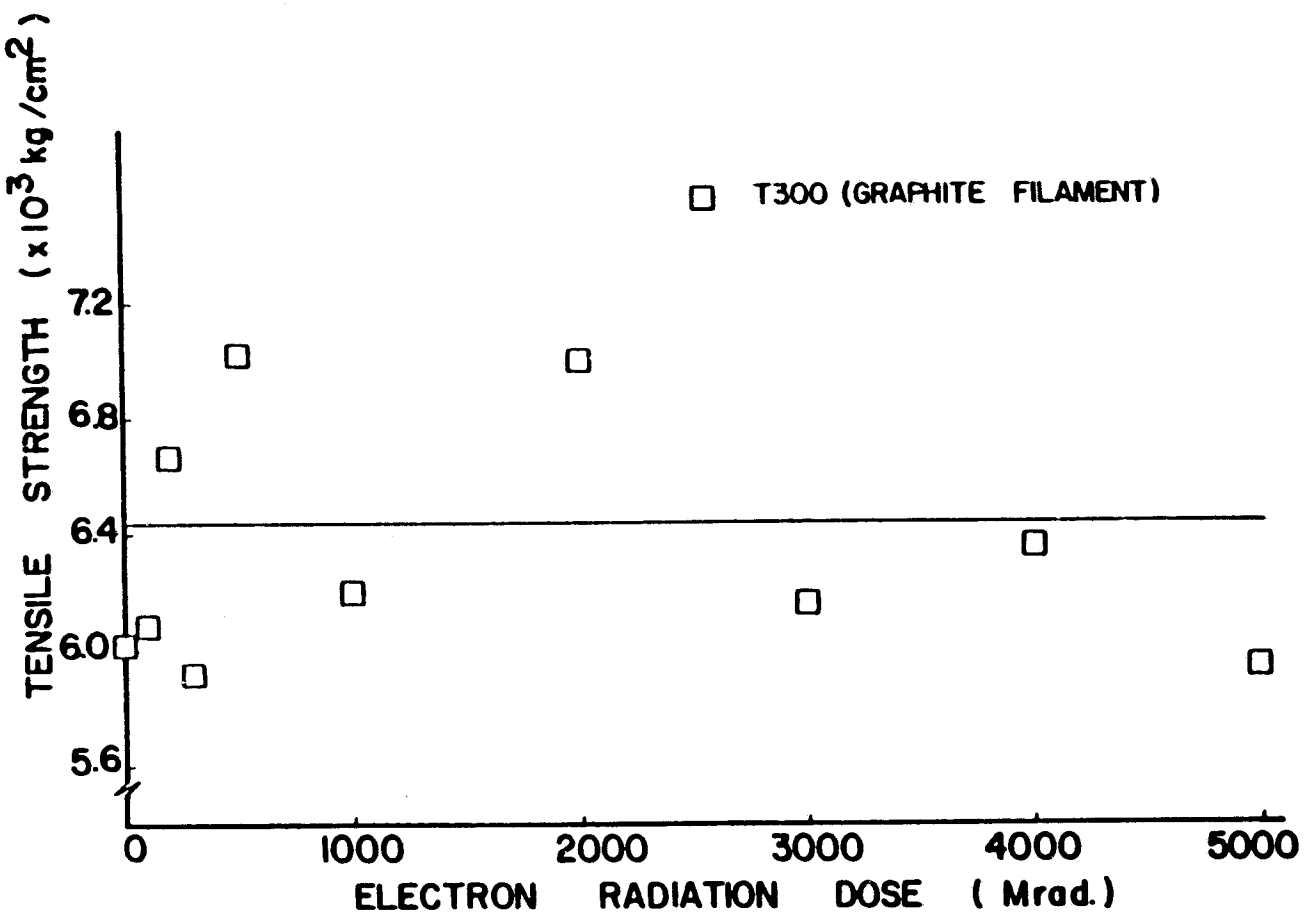


Figure 19. Tensile Strength of T300, Graphite Fiber, as a Function of Electron Radiation Dose.

Table 11. Tensile Strength of C6000 as a Function of Electron Irradiation Dosage

[redacted]	Control	10⁴ Mrad	200 Mrad	300 Mrad	500 Mrad	1000 Mrad	2000 Mrad	3000 Mrad	4000 Mrad	5000 Mrad
Pre-cond. (days)*	14	14	14	14	14	14	14	14	14	14
Post-cond. (days)**	105	105	105	105	105	105	105	105	105	105
No. of specimen	5	5	4	5	5	5	5	5	5	5
Tensile str. (Kg/cm^2)	13300	12830	11520	13440	14180	13610	12950	12920	12940	13230
Standard Deviation	1485	919	1037	856	711	1122	925	1193	1254	302
C.V.%	11.1	7.2	9.0	6.3	5.0	8.2	7.2	9.2	9.8	2.4
Average modulus (kg/cm^2)	227,700	217,800	169,400	229,300	279,200	277,700	244,900	264,800	237,000	221,300

*Pre Condition — 80°C in vacuum desiccator.
**Post Condition — 65% R.H., 20°C.

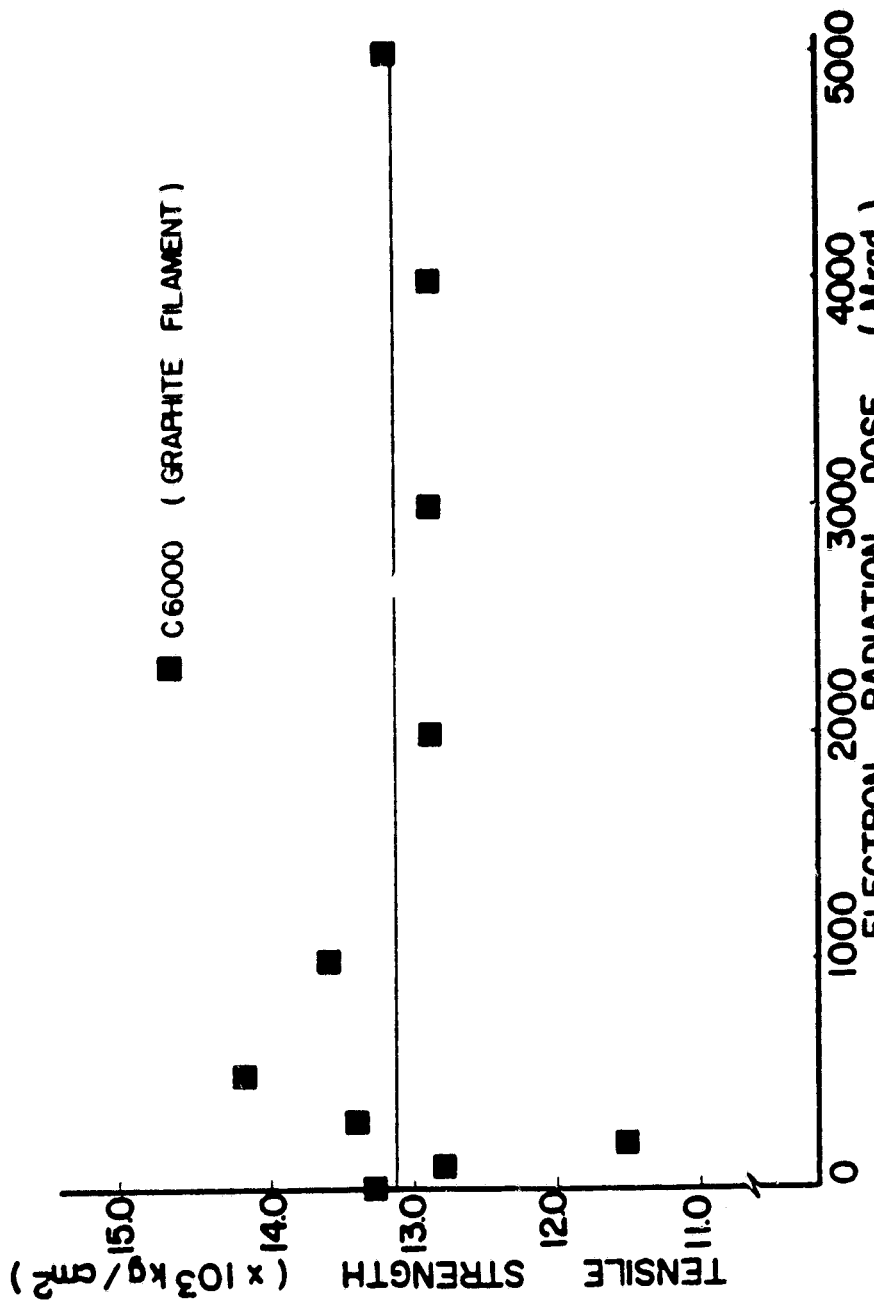


Figure 20. Tensile Strength of C6000, Graphite Fiber as a Function of Electron Radiation Dose.

stress and modulus of the composites is to slightly increase the strength of all types of composites studied. The results reported here are consistent with earlier work on plastics, fibers and composites as discussed below.

Parkinson and Sisman [12] reported that polymers containing aromatic rings were highly resistant to radiation and attributed this resistance to the absorption and dissipation of energy without bond disruption of aromatic rings. For example, the aromatic amine-cured epoxy (diamino diphenyl methane), which was the same composition as NARMCO 5208, retained > 80% of its initial strength after being irradiated to a gamma dose of 1000 Mrad in air.[12] PMR-15, a polyimide containing aromatic rings, was a radiation resistant polymer which retained its strength up to 10^{10} rads (10,000 Mrads) of gamma radiation in vacuum.[12] P1700, an aromatic polysulfone, showed an increase of 15% when irradiated with a gamma dose of 600 Mrad in vacuum at 125°C.[16] From these two references [12, 16], we can see that there are two things which protect polymers from degradation, i.e., the aromatic ring of polymer and the oxygen-free irradiation exposure. All the matrices used in this study contain aromatic rings, and the irradiation exposures were done either in vacuum after prevacuum treatment or after pre-vacuum treatment and without the presence of oxygen. This should have reduced the tendency of radiation damage. In fact, the excitation and ionization resulting from these radiations probably cause additional crosslinking reactions to occur rather than the chain scission in these types of matrices.[19] In effect, the radiation continues the curing process of the composites.

Other support for the increase in stress and modulus of the composites is the effect of radiation on graphite fiber. As noted earlier, Bullock[25] found an increase of 30% of graphite strength after neutron irradiation. His work suggested that there was some rearrangement in the graphite crystal.

The experimental results in this study showed little change in the strength and modulus of T300 and C6000 graphite fiber over 5000 Mrad electron dose. However, the strength and modulus of these fibers were appreciably lower than the corresponding values for the composites and for the values reported by the manufacturer. Reasons for this large difference is unknown but may be due to the testing procedure used or due to changes in the yarns with time.

Even though the response of the matrix and fiber to neutron and gamma radiation is understood, the behavior of the composites is much more complicated. It involves the fiber-matrix interface bonding, which depends on the characteristics of the graphite surface and the chemical nature of the matrix.

5. Summary

Graphite fiber/epoxy (T300/5208), graphite fiber/polyimide (C6000/PMR 15) and graphite fiber/polysulfone (C6000/P1700) composites after being irradiated with 0.5 Mev electron radiation in vacuum up to 5000 Mrad, show increases in stress and modulus of approximately 12% compared with the controls. Graphite fiber/epoxy (T300/5208 and AS/3501-6) after being irradiated with 1.33 Mev gamma radiation up to 360 Mrads, show increases in stress and modulus of approximately 6% at 167 Mrad compared with the controls. Therefore, the results from this study suggest that the graphite fiber composites used in this study should withstand the high energy radiation in a space environment for a considerable time, e.g. over 30 years.

6. Recommendation

For further research, the radiation treatments should be at the higher dosage, until the failure of the composites is reached. This will provide information for the life-time of the composites.

The next consideration is the test method to determine other mechanical properties, such as the horizontal shear strength and interlaminar shear strength,[20,22,36] which give the information about the fiber-to-matrix interaction and the transverse flexural strength[22] which gives information about the matrix.

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